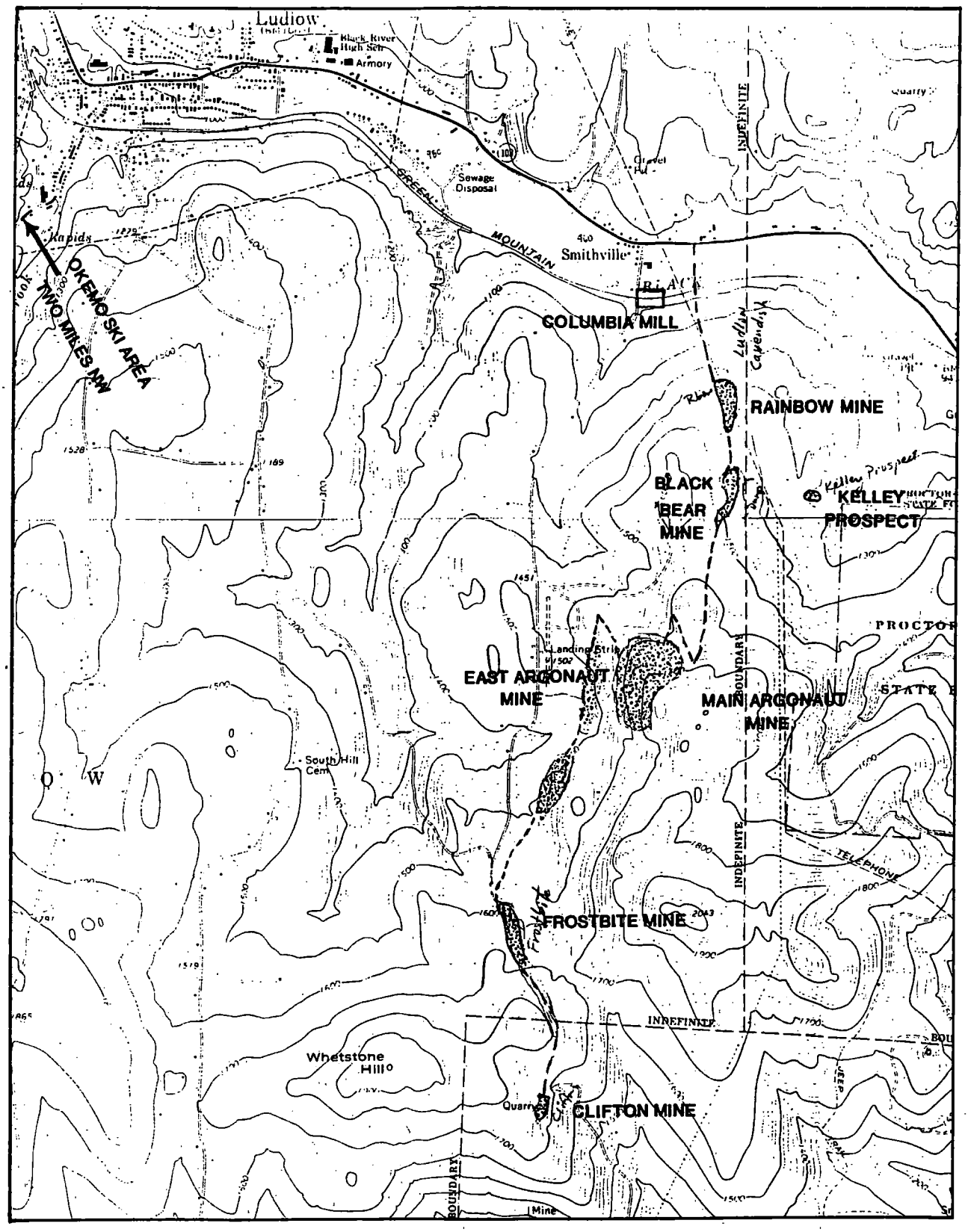


SCALE 1/2 0 1 MILE

TALC TREND

TALC DEPOSIT

CYPRUS LUDLOW AREA
TALC OPERATIONS

Black Bear

Black Bear contains an accessible 300,000 ton reserve of + 75% GEB material. Mining was carried out in 1991. Total reserves are estimated at 1,300,000 tons mineable, of which 300,000 are estimated to be +75% GEB and 700,000 at +65% GEB. With most of its ore directed for Columbia and Chester, Black Bear provides a viable future reserve of high brightness material for industrial products.

Argonaut

The most important reserve on the Ludlow trend is the Argonaut where the Argonaut Main ore body open pit, a three million ton deposit of medium (>65-75 GEB) brightness reserve, has been supplanted by the development of the Argonaut East ore body due to the high stripping ratio and possibly high incidence of fibre bearing zones encountered at the main ore body.

The Argonaut East ore body offers a potential of 3 million to 5 million tons of material that is in the inferred category. Of the eight drill holes directed to it in 1988, only four can be used to estimate the reserve. They, with past mapping in older underground workings, suggest a substantial mineable tonnage here averaging perhaps 20% +75 GEB material. The deposit is currently being developed with stripping and mining of shallow ore. The extension of the Argonaut mining permit for this work has been protested by a nearby homeowner and supporters. The development would be within view of the Okemo ski area, some 4 miles distant.

The Argonaut East ore body presents good potential for a high tonnage of medium to high brightness reserves of Ludlow type ore.

Alpine Alabama

The Alpine deposit is a small zone of talc quartzite whose saprolite reserve was mined by open pit in the past. Current reserve is in hard rock. CIM ore reserve calculation shows a mineable reserve of 75,000 tons in a remaining 100' depth - 80% of this is +80 GEB # 1 ore. The waste to ore ratio is calculated at 5:1.

An independent cross section manual on reserve calculation supports these reserve numbers.

Alpine ore goes to Alpine Mill stockpiles where it is used to produce 3,600 tons of Regal, Act II, Alphafil and YB products using # 1 ore, 33%; # 2 and # 3 ore (40-80% GEB) 33%, see Anderson's chart page 36. Recovery is given at 70%; so ore requirement then will be 3,445 tons per year.

Alpine was campaign mined in 1991 (21,000 tons) and (by inspection) there is at least that tonnage (estimated at +30,000 tons) on the stockpile.

The next mine campaign is planned for 1996. However, pit is flooded and the future accessibility of new ore is impossible to estimate.

The Alpine ore isn't perfect; it needs bleaching and has a high quartz content in some areas. The Alpine mill has and could run without Alpine ore, whose main value is its apparent low cost (given at $30/ton).

From Drew Anderson, John Close, Brychan Griffiths

Date February 14, 1992

To ~~From~~ E. H. Reade

Subject Crude Required to Produce Finished Goods

CIMC PRODUCT LINE	Fin. Goods 1991 TOTAL TONS	CRUDE No. 1	CRUDE No. 2	CRUDE No. 3	CRUDE No. 4	$ VALUE
Regal, Act II, Alphafil YB	3600	Alabama #1 33% @ $30/ton	Alabama #2 17% @ $30/ton	Alabama #3 17% @ $30/ton	Australian Fines (Stained) 33% @ $95/ton	
Altalc	2600	Beaverhead #19 grade 100% @ $175/ton				
Supra Suprafino	2100	Italian 50% at $282/ton	Australian Cosmetic @ $147/ton			
Supra EF A	200	Italian 75% at $282/ton	Beaverhead 25% @ $175/ton			
Top Note Brillante Alphaglide	180	Italian 100% at $282/ton				
Aura Stellar	260	Austr. Cosm. 100% at $147/ton				

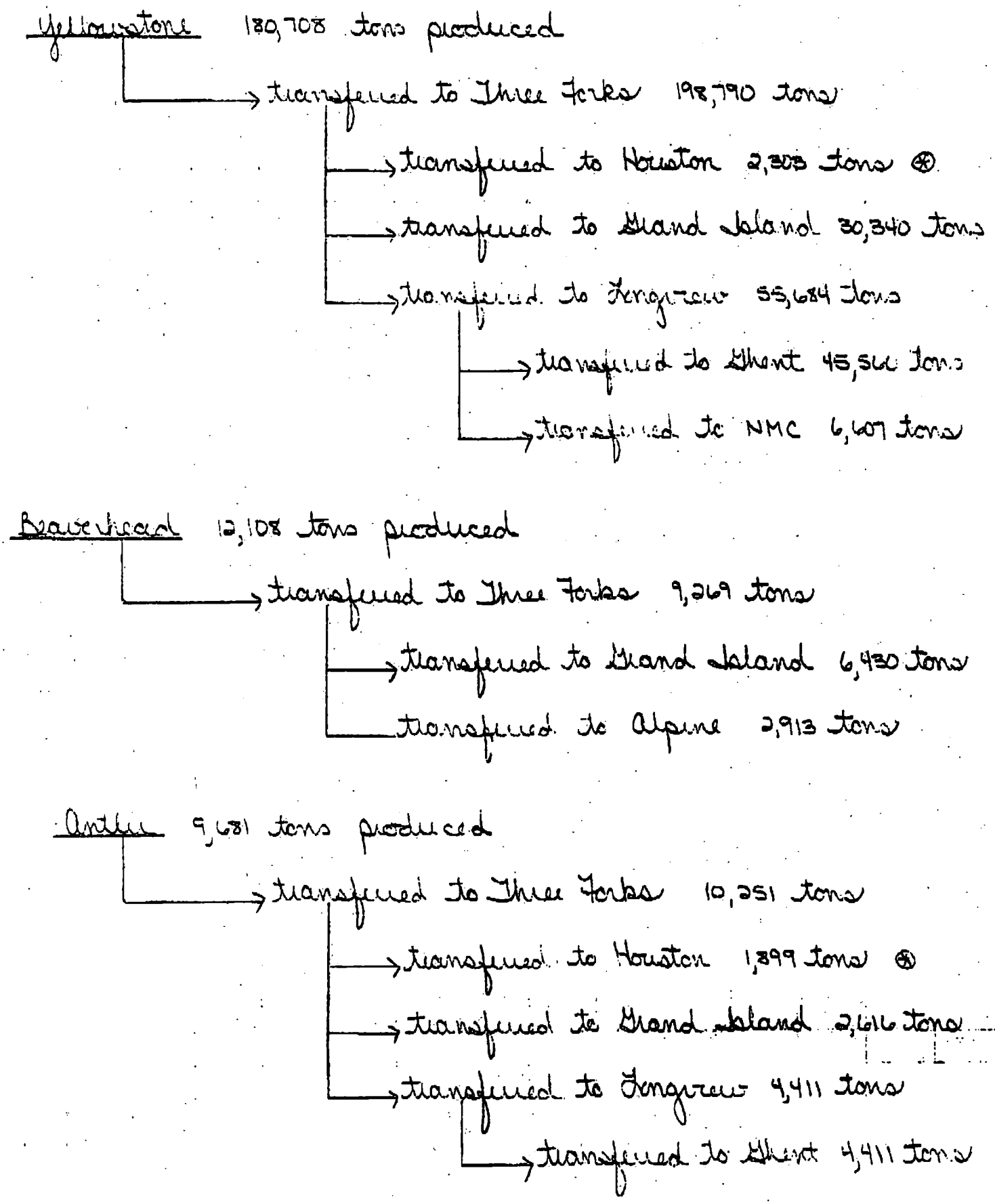
NOTE: Crude blends should show origin, percent & value per ton.
Ex.: 20% BVHD @ $125/T.

PLEASE FAX RESULTS TO YELLOWSTONE ON 2/17.

cc: R. D. Baker
F. F. Boyl
M. J. Lorang
B. Wright

2-15-92

CYPRUS



⊛ ultimately shipped into Mexico.

Hammondsville 351 tons produced

→ transferred to West Windsor 351 tons

Hudson 147,858 tons produced

→ transferred to Columbia 141,086 tons

→ transferred to Chester 5,573 tons

→ transferred to West Windsor 5,658 tons

Hamm 103,927 tons produced

→ transferred to Chester 103,927 tons

→ transferred to Johnson 18,191 tons

→ transferred to West Windsor 37,533 tons

Ivy 45,675 tons produced

→ transferred to Johnson 51,090 tons

Red Hill/Yale City 15,289 tons produced

→ transferred to Tayon 15,289 tons

Alpine Mine 21,062 tons produced

→ transferred to Alpine Mill 902 tons

Malaga Mine ø tons produced

→ transferred to Malaga Mill ø tons

→ transferred to Ghent 2,496 tons

Exhibit G

MAR 25 1992

INTEROFFICE CORRESPONDENCE

LOS ANGELES

TO	SEE DISTRIBUTION	DATE	March 25, 1992
ATTENTION			L.A. FILE
FROM	R. C. MUNRO		YOUR FILE
SUBJECT			COPIES TO

CYPRUS ORE RESERVES - ARSENIC & TREMOLITE

Excerpts from Cyprus Talc Reserve Report by R.C. Munro

Geology & Environment

There are some important environmental issues related to the geology and mineralogy of the Cyprus talc deposits, particularly in Vermont.

Arsenic

Arsenic iron sulphides (arsenopyrite) are, with their alteration products, present in many of the talc-carbonate schist ore zones in the Vermont area. Total arsenic, as analyzed in the Ludlow Rainbow deposit, averages generally less than 100 ppm but with some small zones in excess of 1000 ppm. No apparent major effort is underway to regularly monitor or completely assess the total arsenic content of ores, tailing solids and wastes although the distribution of sulphides and arsenates in the talc ore system is generally understood.

In near surface weathering zones, crushed rock, stock piles and mine working areas, the arsenic sulphides (above) convert in part to the more soluble arsenates, for example, the hydrous nickel arsenate, annabergite (38% AS_2O_5). Soluble arsenic is measured in cores, ore samples, mill feed, product and tailings. Soluble arsenic content is monitored and governed under EPA/OSHA regulations.

High (e.g. +6 ppm As) soluble arsenic contents of mill feed at the West Windsor mill contribute to reduced recoveries and milling rates. At West Windsor, part of the mill recovery problem at least is being ascribed to a high fines content in the feed and to low pH of the process water, both of which contribute to increased soluble As. The problem has been under study at West Windsor since 1987 by Mill Manager, Jeff Scott, who indicated that if the arsenic content is above +6 ppm soluble As and the talc content falls below 62% talc production rates and recoveries can fall by 50%. The product specs are -3 ppm As or less at West Windsor and current material in the silos is measured at 0.73 ppm to 2.33 ppm soluble As.

To me, there also seems to be the overall risk of continuing conversion of As in sulphide to more soluble arsenates in some stockpiles, waste, and solid tailings as acid, water, air and time work on them.

Tremolite

The other serious mineralogical contaminant in the talc ores of Vermont is the fibrous variety of the amphibole minerals, tremolite and actinolite (hydrous calcium iron-magnesium silicates) which have been classified as asbestiform minerals by OSHA and EPA. OSHA was expected to de-classify non-fibrous (blocky) tremolite on February 29, but has not as yet announced their decision.

As a result, all tremolite, the fibrous varieties of all amphiboles and chrysotile asbestos in talc ores are a source of great concern to all talc producers and especially to marketers of cosmetic products.

Cyprus claims that there are no fibres in their cosmetic talc products and they work rigorously to ensure this. However, a recent paper published by Rutgers University worker, Alice Blount, suggests the presence of fibre in several cosmetic talcs, some of which might have been from Cyprus West Windsor material, which is a source of great concern to Cyprus management and potentially to their principal customer, Johnson & Johnson. Talc de Luzenac personnel are well aware of the situation and Phillipe Moreau is currently quietly working to identify the reality and the magnitude of the problem.

Vermont talcs are derived from altered serpentine - a natural host for asbestiform minerals. There is certainly visible tremolite and actinolite in specific zones of the Vermont deposits - fibrous tremolite was identified by the writer in exposures and cores at the East Argonaut and Black Bear mines. Cyprus staff report past tremolite from the Hammondsvile and Clifton deposits.

Tremolite in these deposits is encountered in the contact zones between the talc and the surrounding schist; in "grey talcs" in the vicinity of the contacts; and associated with the chlorite/amphibole waste zones within the talc ores that are locally termed "cinders". Cyprus maintains a selective mining program in Vermont that is directed toward exclusion of all of these potentially fibre-bearing zones from the ores sent to the mills, and those suspect tonnages, including the associated talc, are left in the pit walls or sent to waste piles.

Minor occurrences of amphiboles and asbestiform minerals are also attributed to confined areas of the Montana deposits. Tremolite (blocky) was encountered in a dike zone at Antler. A chlorite zone at intersecting faults at Yellowstone S40 contained some minor tremolite, and stockpiles of Beaverhead open pit fines, slated

for burial, have been measured at 0.33% to 0.70% tremolite by Three Forks and Alpine Mill Labs.

No fibrous material showed up in samples taken by the writer at the Western Source Red Hill mine in California, but minor tremolite is possibly present in the contact zone where it should be avoidable by selective mining.

Arsenic content (total and soluble) and the presence of fibrous minerals in exposed stockpiles and waste need to be checked at Alpine, Alabama and the now closed California properties operated by Cyprus in the past.

/eji

DISTRIBUTION:

R. J. Kerstetter
G. L. Toll
G. B. Lawson - BCL
J. Paulsen
P. Moreau - Talc de Luzenac

Exhibit H

GMW

Alice M. Blount, Ph.D.

Mineralogist

April 23, 1998

M. Raymond Hatcher
MEHAFFY & WEBER
2615 Calder Avenue
P.O. Box 16
Beaumont, Texas 77704

Dear Mr. Hatcher:

According to your letter of March 31, 1998, I have written and enclosed a report on the occurrence, regulation and up-to-date scientific view of asbestos, amphiboles and "intermediate" fibers. I have also enclosed copies of my 1990 and 1991 papers, one of which I am sure that you already have. The 1991 paper was written because I became aware that it was a common opinion among industrial hygienists that industrial talcs were better than pharmaceutical and cosmetic talcs because there was a regulation for the former and not for the latter. I knew that this was not the case and wanted to set the record straight.

Although my papers report an improved method for analysis, the determinations for the sample labeled ***I*** (Johnson & Johnson's Vermont talc) have been done by the traditional methods as well (see Table 2, page 567 in the 1990 paper). As I told you, I believe that Johnson & Johnson's Vermont talc contains trace amounts of asbestos which are well below those specified by OSHA. It should be noted that the proposed FDA regulation, which was never finalized, also specified the same 0.1% limit for amphibole asbestos as OSHA.

I may be away for short periods during the coming weeks, but I do check for messages on my work phone at the number you have been using.

Sincerely yours,

Alice M. Blount

Alice M. Blount, Ph.D.

Box 3437
Rutland, VT 05701
Phone: 802-747-4857

e-mail: amblount@together.net

EXHIBIT H

J&J-0049150

JNJ 000064086

Exhibit I

Luzenac AMERICA

Luzenac America Technical Center • 8985 East Nichols Avenue • Englewood, CO 80112 • (303) 643-0451 • Fax: (303) 799-8926

TECHNICAL REPORT

To: **David Crouse**

Analytical Project No: **A01709**
Date: **23-May-02**

From: **Julie Pier**
Analytical and Technical Support

Copy: **J. M. Godla**
S. S. Mauney
R. J. Zazenski

Subject: **ANALYSIS OF FIBROUS MATERIAL FROM ARGONAUT WASTE ROCK**

Request:

A sample of fibrous material from the waste rock on the west side of the south end of the Argonaut mine was submitted to the Technical Center for identification. The waste rock was being considered for road paving applications.

Results:

The fibrous material is tremolite.

The material was first examined by polarizing light microscopy, using the dispersion staining technique. Tremolite was preliminarily identified by this method.

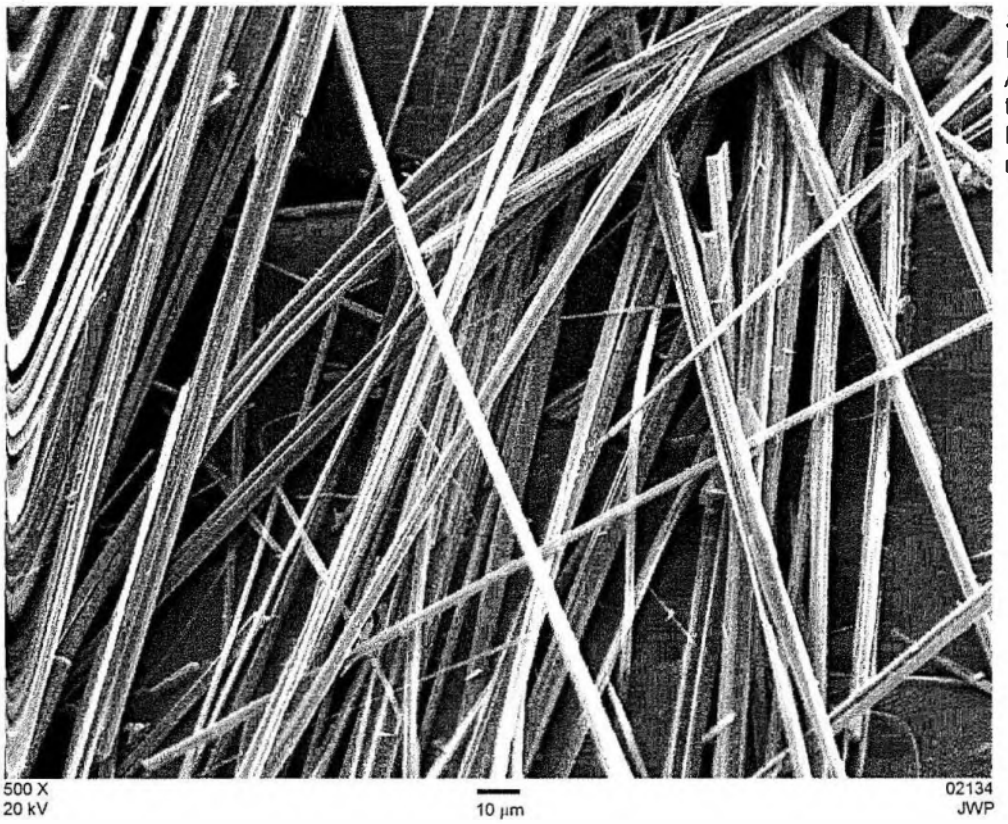
Subsequent analysis by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) confirmed the tremolite identification. SEM micrographs and chemical analysis by energy dispersive X-ray spectroscopy (EDS) are included in Plate 1.

Plaintiffs' Exhibit IC-420

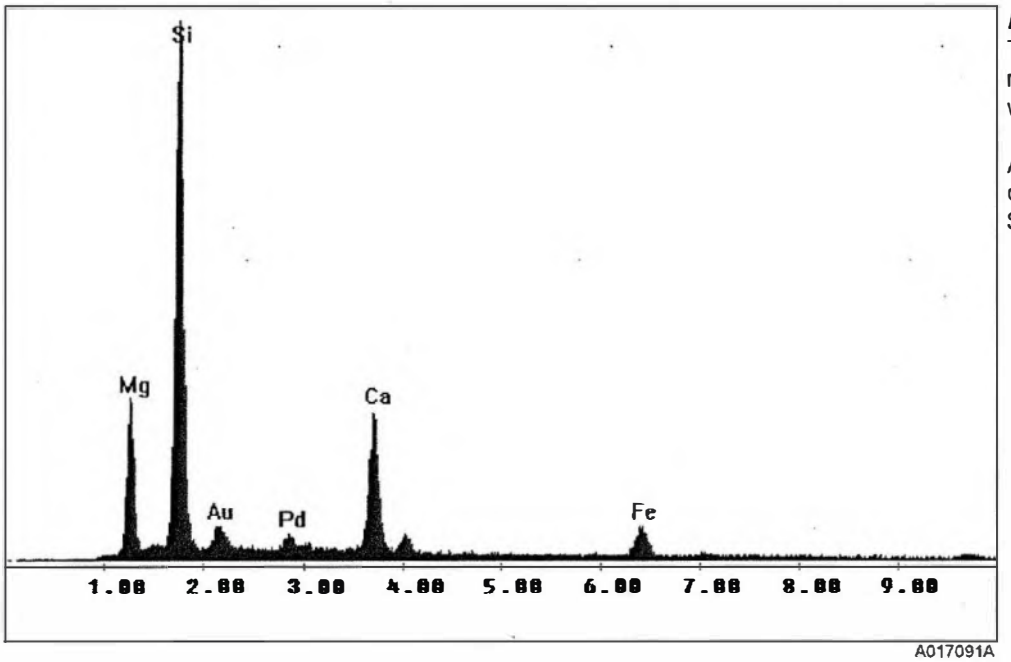
ANALYSIS OF FIBROUS MATERIAL FROM ARGONAUT WASTE ROCK
Project No. A01709

Plate 1

LUZENAC AMERICA TECHNICAL CENTER
23-May-02
J.W. Pier



SEM IMAGE
Fibrous material found in Argonaut waste rock identified as tremolite. The material clearly has an extremely high aspect ratio.



EDS CHEMICAL ANALYSIS
The chemical analysis of the material, above, is consistent with tremolite.

Au and Pd peaks are from a conductive coating applied for SEM analysis.

Exhibit J

FEB 24 2004 5:49 PM FR J-J CORP PR 732 524 2153 TO 919089043738 P.01

facsimile transmittal

To: Steve Mann - CPC Fax: (908) 904-3738

From: Marc Monseau Date: 2/24/2004

Corporate Communications

Re: Asbestos Pages: 4

CC:

☐ Urgent ☐ For Review ☐ Please Comment ☐ Please Reply ☐ Please Recycle

Steve,

Last week, we were contacted by a reporter at a Sacramento television station who wanted to get our reaction to a test they performed on Johnson's Baby Powder. She has since sent me the attached cover letter and lab results, which Sarah Colamarino suggested I share with you.

Can you please review? Sarah will be calling you shortly to discuss. In the meantime, if you have any questions, please give me a call.

Best regards,

Marc Monseau

Corporate Communications

(732) 524-1130

PLAINTIFF'S TRIAL EXHIBIT 2843

EXHIBIT J

FEB 24 2004 5:49 PM FR J-J CORP PR 732 524 2153 TO 919089043738 P.03
02/23/04 MON 11:47 FAX

KCRA-TV3

3 Television Circle Sacramento CA 95814 916 446.3333

February 23, 2004

To: Mr. Mark Monseau
Johnson & Johnson

From: Millicent Ozdaglar
KCRA TV3

Greetings Mr. Monseau: Thank you for taking my call last week regarding KCRA TV3's Special Report on asbestos. This is a working story with no airdate. Our reporter/anchor Dave Walker is investigating the existence of harmful levels of asbestos in our community. One of the elements of the story takes a look at asbestos in household products and building materials.
One of the items tested by Hayward Laboratory was Johnson's baby powder, which tested at above normal levels for asbestos.
I am enclosing a copy of the test and results for you to look over. If you could please give me a call once you have reviewed the material, I would like to talk about the results with you.

Sincerely,

Millicent Ozdaglar
KCRA TV3 Special Projects Producer
3 Television Circle
Sacramento, Ca. 95814
(916) 325-3288

Forensic Analytical

QUANTITATIVE ANALYSIS REPORT
ASBESTOS IN BULK MATERIAL
Transmission Electron Microscopy*

Michael Bowker
4069 Alice Ct
Placerville CA 95667

Date Collected:
Job ID: KCRA Television/Dave Walker
Site:

Page:	1 of 1
Client Number:	A30388-1
Report Number:	T006525
Date Received:	12/19/03
Analyst:	RE
Date Analyzed:	1/5/04
Date Reported:	1/5/04

Sample Preparation: Each sample was prepared using the following gravimetric techniques. Representative subsamples were weighed, ashed for >12 hours, at 480C, and reweighed to determine the organic proportion. The ashed residues were grated in concentrated hydrochloric acid, dried, and reweighed to determine the acid-soluble component weight percent. The acidified residue was resuspended in a known volume of particle-free water and sonicated. Aliquots of the suspension were brought to ~20ml and filtered through 0.22um pore-size mixed cellulose ester (MCE) membranes. After air-drying, these membranes were collapsed, etched, carbon-coated, and mounted on 200-mesh copper TEM grids.

Analytical Method: The analysis was performed on a Philips CM12 or Hitachi H600AB TEM at 100kV accelerating voltage. An extended low magnification analysis (~2,500x) was performed for large asbestos structures, followed by a high magnification analysis (~19,000x) for smaller asbestos structures. Asbestos structures were identified by morphology (Yamate Level II definitions), qualitative selected area electron diffraction (SAED), and energy dispersive x-ray analysis (EDX). In addition, the length and diameter of each asbestos structure were recorded.

Data Reduction: The asbestos concentration in each sample was calculated by first determining the volume of each asbestos structure counted, and then using magnification and density conversion factors to determine asbestos mass. The mass detected in the high magnification analysis was then normalized to the number of grid openings analyzed and the aliquot volume filtered for the low magnification analysis. Since a known residue mass was passed through a known filter area, and the filter area analyzed is also known, the normalized asbestos mass in the residue can be determined and then back-calculated to the weight percent asbestos in the original sample.

ANALYTICAL RESULTS

Client Sample Number	Lab Sample Number	Organic Weight Percent	Acid-Soluble Weight Percent	Asbestos Weight Percent	Asbestos Type(s)**	Residue Weight Percent
TEM-01 (Johnson's baby powder)	20025738	3.6%	6.7%	0.20%	AN	89.3%
TEM 03 (Revlon Blush)	20025739	28.7%	13.1%	<0.0001%	ND	57.2%

Mark S. Haye, EM Supervisor, Hayward Laboratory

* EPA Test Method 600/R-93/116, Part 2.5: Method for the Determination of Asbestos in Bulk Building Materials.

** Asbestos types: CH=chrysotile; AM=amosite; TR=tremolite; AC=actinolite; CR=crocidolite; AN=anthophyllite; ND=none detected.

Sample Preparation: Each sample was prepared using the following gravimetric methodology: representative subsamples were weighed, ashed for >12 hours, at 400C, and reweighed to determine the organic proportion. The ashed residues were ground in concentrated hydrochloric acid, dried, and reweighed to determine the acid-soluble component weight percent. The acidified residue was resuspended in a known volume of particle-free water and sonicated. Aliquots of this suspension were brought to >20kPa and filtered through 0.22um pore-size mixed cellulose ester (MCE) membranes. After air-drying, these membranes were collapsed, etched, carbon-coated, and mounted on 200-mesh copper TEM grids.

Analytical Method: The analysis was performed on a Philips CM12 or Hitachi H600AB TEM at 100kV accelerating voltage. An extended low magnification analysis (~2,300x) was performed for large asbestos structures, followed by a high magnification analysis (~19,000x) for smaller asbestos structures. Asbestos structures were identified by morphology (Yamate Level II definitions), qualitative selected area electron diffraction (SAED), and energy dispersive x-ray analysis (EDX). In addition, the length and diameter of each asbestos structure were recorded.

Data Reduction: The asbestos concentration in each sample was calculated by first determining the volume of each asbestos structure counted, and then using magnification and density conversion factors to determine asbestos mass. The mass detected in the high magnification analysis was then normalized to the number of grid openings analyzed and the aliquot volume filtered for the low magnification analysis. Since a known residue mass was passed through a known filter area, and the filter area analyzed is also known, the normalized asbestos mass in the residue can be determined and then back-calculated to the weight percent asbestos in the original sample.

ANALYTICAL RESULTS

Client Sample Number	Lab Sample Number	Organic Weight Percent	Acid-Soluble Weight Percent	Asbestos Weight Percent	Asbestos Type(s)**	Residue Weight Percent
TEM-02 (Johnson's baby powder)	20025738	3.8%	6.7%	0.20%	AN	89.3%
TEM 03 (Revlon Blush)	20025739	29.7%	13.1%	<0.0001%	ND	57.2%

Mark S. Floyd, EM Supervisor, Hayward Laboratory

* EPA Test Method 600/R 93/116, Part 2.5: Method for the Determination of Asbestos in Bulk Building Materials.

** Asbestos types: CH=chrysotile; AM=amosite, TR=tremolite; AC=actinolite; CR=crocidolite; AN=anthophyllite, ND=none detected.

3777 Depot Road, Suite 409, Hayward, California 94545-2761 • Telephone 510/887-8828 (800/227-1445) Fax: 510/887-4218

Exhibit K

AMA Analytical Services, Inc.
Focused On Results.

CERTIFICATE OF ANALYSIS

Chain of Custody: 308006
Client: US Food & Drug Administration
Address: Office of Cosmetics & Colors
4300 River Road
College Park, MD 20740
Attention: John Gasper

Job Name: Task 3 - Analysis of Official Samples
Job Location: 4th Group - 15 Samples
Job Number: CLIN 1- Task 3
PO Number: HHSF223201810337P

Date Submitted: 7/24/2019
Date Analyzed: 8/20/2019-9/18/2019
Report Date: 10/3/2019
Date Sampled: Not Provided
Person Submitting: Goran Periz
Revised: 10/11/2019 (Revision #2)

SUMMARY OF ANALYSIS

AMA Sample ID	Client Sample ID	TEM LOD Using ASTM D5756 Mass Calculation	TEM LOQ Using ASTM D5756 Mass Calculation	% Tremolite by TEM Using ASTM D5756 Mass Calculation	% Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Total Tremolite & Chrysotile by TEM Using ASTM D5756 Mass Calculation	% Asbestos by PLM	% Organics	% Acid Soluable	% Other	Comments
308006-6	D-58	0.00000169%	0.00000675%	ND	ND	ND	ND	0.3%	6.7%	93.1%	Organics = 0.3%; Acid Soluable = 7.1%; Other = 92.6% Gravimetric Loss from PLM Prep:
308006-6A	D-58	0.00000133%	0.00001485%	ND	< 0.00001%	< 0.00001%	ND	0.2%	19.5%	80.2%	
308006-6B	D-58	0.00000135%	0.00000540%	ND	0.00002%	0.00002%	ND	0.2%	11.2%	88.6%	Other = 94.2%

LOD = Limit of Detection **LOQ** = Limit of Quantification **ND** = Not Detected **PLM** = Polarized Light Microscopy **TEM** = Transmission Electron Microscopy

Analytical Method(s): PLM by Modified NY ELAP 198.6
TEM by Modified NY ELAP 198.4/ASTM D5756

Analyst(s): PLM
TEM

Technical Director: Andreas Saldivar

All results are to be considered preliminary and subject to change unless signed by the Technical Director or Deputy

EXHIBIT K
JNJTALC001284317
Prudencio Pltfs' Ex. 1571 pg1

Record Changes Report

Client: US Food & Drug Administration

Client Code: FDA

Chain of Custody: 308006

Date	Description
10/11/2019	308006 6, 6A, 6B/D 58: 1) added initials & dates to all strike throughs and additions to gravimetric bench sheets. 2) revised handwritten TEM bench sheet for 6B to break up the single cluster found on Grid B, GO I8 into its 3 component fibers 3) changed the word "fiber" to "structure" on p. 4 of Case Narrative under LoQ discussion for 6A & 6B & updated the basis of LoQ calculation for 6B. 4) changed the word "fiber" to "structure" in reference to chrysotile on p.4 of Case Narrative under the TEM Discussion and Interpretation of Analytical Findings. 5) Updated the picture for 308006 6B Chrysotile Structure 1 on p. 6 of Case Narrative. 6) revised reported LoQ, concentration of chrysotile & total cocentration for aliquot 6B based off of 4 structures (original concentration was based off of 2 structures). 7) added gravimetric loss data for PLM preparations to comments section of the certificate of analysis.
10/08/2019	308006 6, 6A, 6B/D58: 1) The Special Instructions section of the login sheet was revised to include the FDA's cancellation of a request for analyzing a 4th aliquot of D 58 (308006 6C). 2) The preparation date was added to pages 2 & 3 of the TEM gravimetric bench sheet and to page 2 of the PLM gravimetric bench sheet; an explanation for the date written in the right hand margin of both sets of bench sheets was added to them; added missing weights for 308006 16 and 308006 17. 3) The handwritten TEM Bench Sheet for 308006 6A was revised to explain that the 2nd Chrysotile structure was identified based upon tubular morphology; also the structure number count for the 2nd listed stricture was corrected to read "#2"

AMA Analytical Services, Inc.
Focused on Results www.amalab.com
AIHA-LAP (#100470) NVLAP (#101143-0) NY ELAP (10920)
4475 Forbes Blvd. • Lanham, MD 20706
(301) 459-2640 • (800) 346-0961 • 459-2643

CHAIN OF CUSTODY

(Please Refer To This Number For Inquires) 308006

Information:

1. Client Name:
2. Address
3. Address
4. Address 3:
5. Phone #: Fax #:

4. Contact Perso : Cell:
5. Cell:

Reporting Info (Results provided as soon as technically feasible). If no TAT/Reporting Info is provided, AMA will assign defaults of 5-Day and email/fax to contacts on file.

AFTER HOURS (must be pre-scheduled)
❑ 4 Hours ❑ Late Night
❑ Immediate Date Due:
❑ 24 Hour. Time Due:
Comments:

NORMAL BUSINESS HOURS
❑ 4 Hours
❑ Same Day
Next Day
❑ 2 Day
❑ 3 Day
❑ 5 Day +
Date Due:
❑ Results Required By Noon

REPORT TO:
❑ Email:
❑ Email 2:
❑ Verbals:

Asbestos Analysis
*P M Air Please Indicate Filter Type:
❑ NIOSH 7400 (QTY)
❑ Fiberglass (QTY)
TEM Air* – Please Indicate Filter Type:
❑ AHERA (QTY)
NOSH 7402 (QTY)
❑ (specify) (QTY)
P 1 Bulk
❑ EPA 600 – Visual (QTY) ❑ Pos Stop
❑ Point Count
❑ Y State Friable 198.1 (QTY)
Grav Reduction ELAP 198.6 (QTY)
Other (specify) (QTY)
MISC
❑ Asbestos Soil PLM__(Qual) PLM__(Quan) PLM/TEM__(Qual) PLM/TEM__(Quan)
*It is recommended that blank samples be submitted with all air and so samples

TEM Bulk
ELAP 198.4/ tatfield
State PLM/TEM

TEM
❑ Qual. (pres/abs) Vacuum/Dust_____ (QTY)
Quan. (s/area) Vacuum D5755-95 (QTY)
❑ Quan. (s/area)Dust D6480-99 (QTY)

TEM Water
Qual. (pres/abs) (QTY)
❑ ELAP 198.2/EPA 100.2 (QTY)

Analysis
❑ Pb Paint Chip (QTY)
❑ *Pb Dust Wipe (wipe ype _____(QTY)
❑ Pb Soil/Solid (QTY)
❑ Pb TCLP (QTY)
Drinking Water❑ Pb_____(QTY) ❑ Cu_____(QTY) ❑ As_____(QTY)
❑ Waste Water❑ Pb_____(QTY) Cu_____(QTY) As
❑ Pb Furnace (Media) (QTY)

Fungal Analysis
Collection Apparatus for Spore Traps/Air Samples:
Coll ction Media
❑ *Spore Trap_____ (QTY) ❑ Surface Vacuum Du t_____ (QTY)
❑ *Surface (QTY)
❑ *Surface Ta (QTY)
❑ Other (Specify_____)_____ (QTY

SAMPLE I FOR IATIO			ANALYSIS	ULK MATRIX	COMMENTS /
CLIENT ID #	SAMPLE LOCATION/ ID	DATE/	VOL (L)/		SPE IAL IN TRUCTIONS

Print Name Date Time

ATTACHMENT B: CFSAN OFFICE OF COSMETICS AND COLORS CHAIN OF CUSTODY FORM

CF AN
Office of Cosmetics and Colors
CHAIN OF CUSTODY FORM

Case/Lab

Submitter: ___Goran Periz

Assignment No./ Contract No.: HHSF223201810337P

Date Sealed: 7/23/2019 Sample Type: 15 samples D-53 to D67

4

6

9

10

11

12

13

14

Chain of

Item #	Date	Received by (Print	Received by	Comments/Location
1-15	7/24/2019			

CHAIN OF CUSTODY FORM
(Continued)

Final
Authorization for Disposal

Item(s) #: on this document is/are no longer needed as evidence and is/are authorized for disposal by (check appropriate disposal method)

☐ Return to Submitter ☐ Destruction

Name of Authorizing Official: Date:

Signature:

Witness to Destruction of Evidence

Item(s) #: on this document were destroyed by (Name)
in my presence on (date)
Name of Witness to destruction: Signature: Date:

Adapted from: Technical Working Group on Biological Evidence Preservation. *The Biological Evidence Preservation Handbook: Best Practices for Evidence Handlers*. U.S. Department of Commerce, National Institute of Standards and Technology. 2013.

Release to Lawful Owner

Item(s) this document was/were released by Evidence Custodian
ID#: to

Zip Code:
Telephone Number: (_____)
Under penalty of law, I certify that I am the lawful owner above

Signature: Date:

Copy of Government-issued photo identification is attached. ☐ Yes ☐ No

This is to be retained as a permanent record by the Center for Food Safety and Applied Nutrition, Office of Cosmetics and Colors.

Adapted from: Technical Working Group on Biological Evidence Preservation. *The Biological Evidence Preservation Handbook: Best Practices for Evidence Handlers*. U.S. Department of Commerce, National Institute of Standards and Technology. 2013.

AMA Analytical Services, Inc.
Focused On Results.

NVLAP Lab Code 101143-0
NY ELAP Lab ID 10920

Case Narrative

Client Name:	FDA Office of Cosmetics & Colors	Contact:	John Gasper
PO Number:	HHSF223201810337P	Phone:	(240) 402-1133
Job Name/Location:	Task 3 – Analysis of Official Samples (4th Group – 15 Samples)	Email:	
AMA COC Number:	308006-6, 6A, 6B/D-58	Date Received:	July 24, 2019

AMA Sample No.	Client Sample No.	Sample Description	Analytical Method
308006-6	D-58	Slightly clumpy, white powder with a matte appearance	Mod. PLM ELAP 198.6 /TEM ELAP 198.4
308006-6A	D-58		Mod. PLM ELAP 198.6 /TEM ELAP 198.4
308006-6B	D-58		Mod. PLM ELAP 198.6 /TEM ELAP 198.4

Requested Analyses: PLM and TEM Analysis for asbestos fibers conducted by Modified NY ELAP Method 198.6 and Modified NY ELAP Method 198.4

Sample Receipt:
The samples were received by AMA Analytical Services, Inc. on July 24, 2019 at 1058 via in-person drop-off by FDA representative, Goran Periz. The set consisted of 15 (fifteen) samples submitted in ~2oz, glass jars sealed with scotch tape. Conditions were checked upon receipt and all sample containers were intact. Most jars were filled approximately ½ to ¾ full. The sample set was processed on AMA Chain-of-Custody (COC) number 308006. This COC number served as the internal laboratory job number for tracking purposes. The samples were entered into the AMA laboratory database on August 12, 2019 at 1151 by [redacted]. The samples were logged in for analysis in triplicate and each sample aliquot was assigned a unique laboratory identification number as shown in the table above. After the sample login, the set was transferred to AMA's lock-box for storage.

The following pictures document the condition of each sample upon receipt at AMA:

Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

308006-6 6A

D58

Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

Sample Preparation

Samples were prepared for PLM and TEM bulk analysis by (b) (6) on August 13, 2019 through September 5, 2019. Sample preparation consisted of the following steps:

1) Label and weigh two 8mL glass vials for each sample in the set – one vial for the PLM preparation and one vial for the TEM preparation.
2) Weigh out 0.1 to 0.8 grams of material and place in corresponding 8mL glass vial. Record weight.
3) Burn samples at 480° C for at least 12 hours.
4) Record Post-Ash Weight.
5) Treat ashed sample with concentrated hydrochloric acid.
6) Filter acid reduced material onto a pre-weighed 47mm 0.4um PolyCarbonate filter.
7) Place filter into drying oven for 30 minutes and then record Post-Acid Reduced weight.
8) Make four PLM slide preparations from the PLM residual ash for each sample in 1.550 dispersion oil. Make additional preparations in 1.605, 1.625, 1.680 and 1.700 dispersion oil as necessary for particle identification.
9) Weigh a portion of the residue from the TEM residual ash and place it into the corresponding pre-weighed 100ml jar.
10) Fill the 100ml jar with deionized water
11) Sonicate the jars for approximate 5-minutes.
12) Filter 0.2ml to 1ml of the solution onto a 47mm 0.22um MCE filter.
13) Dry the filter for 10 minutes then collapse, carbon coat, and place on a 3 TEM grids.

PLM Analysis

Analysis was performed in accordance with NY ELAP 198.6 protocols. The analysis was conducted using an Olympus BH-2 polarized light microscope (PLM) equipped with a dispersion staining objective. All four slide preparations for each aliquot were examined. 400-point count was performed for those samples on which asbestos was observed. If no asbestos was detected on any of the slides, the percentage of fibrous components was determined by visual estimation. The results of this analysis are detailed below in the *Discussion and Interpretation of Analytical Findings* section for each individual sample.

TEM Analysis

Analysis was performed in accordance with modified NY ELAP Method 198.4 protocols. The analysis was performed using a JEOL JEM-100CX II transmission electron microscope (TEM), equipped with a Thermo Fisher Quest Energy Dispersive X-Ray Analyzer (EDXA), at magnifications of 19,000x. Two grids for each aliquot were examined. Twenty (20) grid openings were examined per sample.

Modifications to the NY ELAP 198.4 Method were:

1) The residue was not placed in alcohol and prepared using the quick drop method. To obtain a more uniform preparation, the residue was placed in a jar and filled with 100ml of deionized water. The jar was sonicated, and a portion of the solution was filtered onto a 47mm 0.22um MCE filter.
2) The tremolite and chrysotile were not visually estimated. The length and width of the observed particles were measured, and the mass of each amphibole particle was calculated using the ASTM D5756 method.
3) All particles identified as tremolite were included with the counts/concentrations, regardless of size and aspect ratio.

The results of this analysis are detailed below in the *Discussion and Interpretation of Analytical Findings* section for each individual sample.

Calculations

ASTM D5756 Mass

$M = \pi/4 \; L * W^2 * D * 10^{-12}$

M = mass
L = length

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W = width
D = density
Percent Calculation

$$\frac{EFA(mm^2) * 100ml * MA(g) * RW(g)}{VF(ml) * IW(g) * AA(mm^2) * RJ(g)}$$

The calculated value is then multiplied by 100 to convert it to percent.

EFA – Effective filter area
MA – Mass of asbestos
RW – Weight of residue
VF – Volume filtered
IW – Initial weight of the sample
AA – Area analyzed
RJ – Weight of residue placed into the jar

Limit of Detection and Quantification
We used the mass of a 0.5 x 0.04-micron tremolite or chrysotile fiber, depending on what was found in each sample, as the basis for our calculations. Limit of detection was defined as 1 fiber and limit of quantification was defined as 4 fibers.

Some aliquots of sample D58 contained very small amounts of asbestos that were either at or below our 4-fiber limit of quantification. For these samples we defined our limit of quantification as follows:

308006-6A: mass of the two observed chrysotile structures plus the mass of two chrysotile fibers measuring 0.5 x 0.04 microns
308006-6B: mass of 4 chrysotile fibers measuring 0.5 x 0.04-micron

Discussion and Interpretation of Analytical Findings:
308006-6, 6A, 6B Client Sample D-58
PLM
All three aliquots of sample D-58 were analyzed by (b) (6) on September 13, 2019. No asbestos or non-asbestos amphibole variants were detected the samples. The results were calculated using the equations detailed in the calculations section.

308006-6	NAD
308006-6A	NAD
308006-6B	NAD

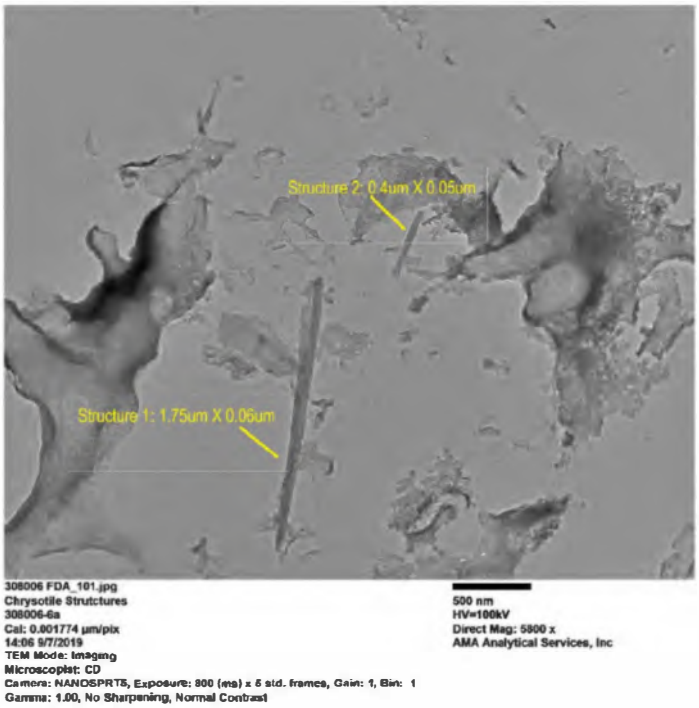
TEM
Sample 6 was analyzed by (b) (6) on September 3, 2019. Samples 6A and 6B were analyzed by [redacted] on September 7, 2019. The primary particle observed was talc along with a few talc fibers, talc ribbons and mica particles. Two Chrysotile structures were detected on the aliquot for 6A and four chrysotile structures were detected on the aliquot for 6B. The results were calculated using the equations detailed in the calculations section.

308006-6	NAD
308006-6A	<0.00002%
308006-6B	0.00002%

Below are pictures, diffraction patterns, and chemistry from some of the observed particles. The unidentified peaks in chemistry spectra are copper, zinc, and carbon. Those peaks are from the TEM specimen holder and specimen grid.

Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

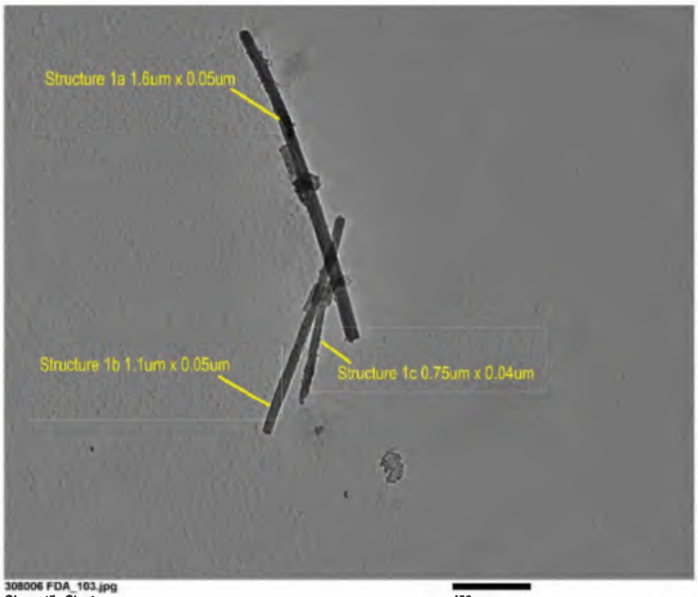
Sample 308006-6A, Chrysotile Structures



Diffraction Pattern from Chrysotile Structure 1 pictured above

Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

Sample 308006-6B, Chrysotile Structure 1



Diffraction Pattern from Chrysotile Structure pictured above

Re: FDA Office of Cosmetics & Colors
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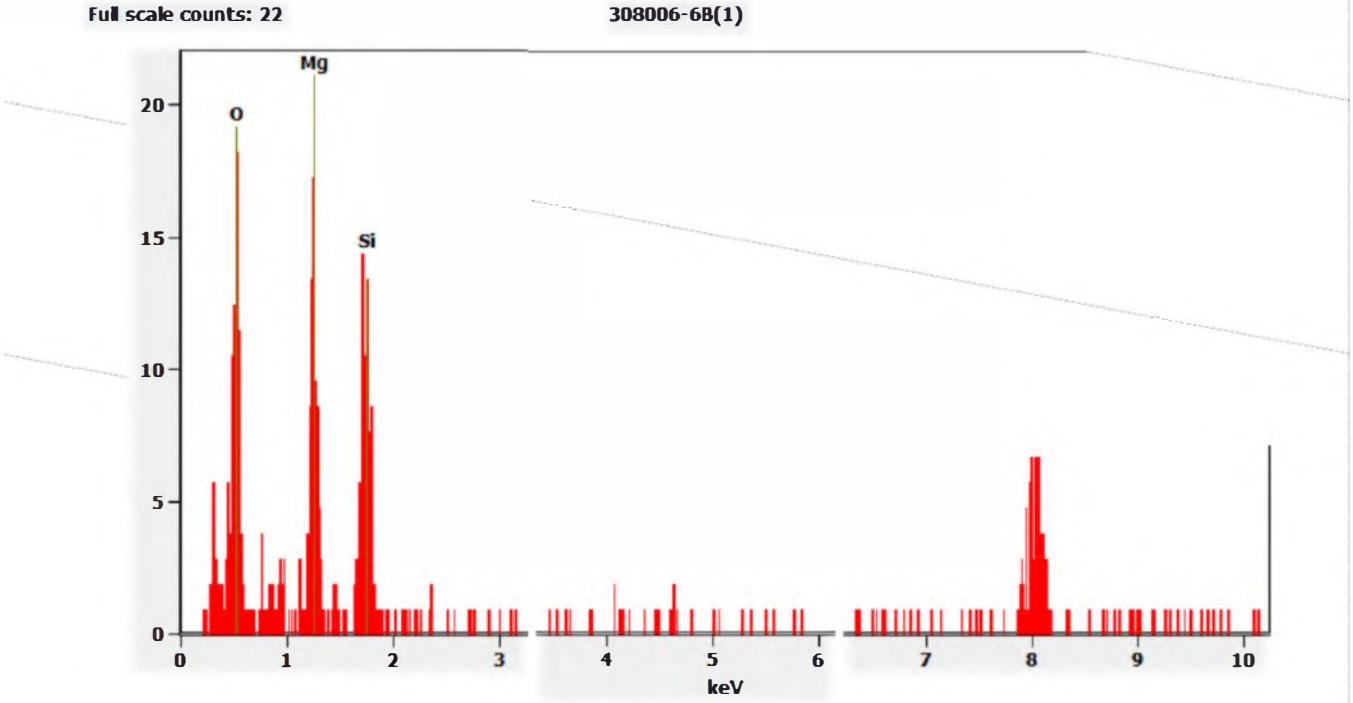
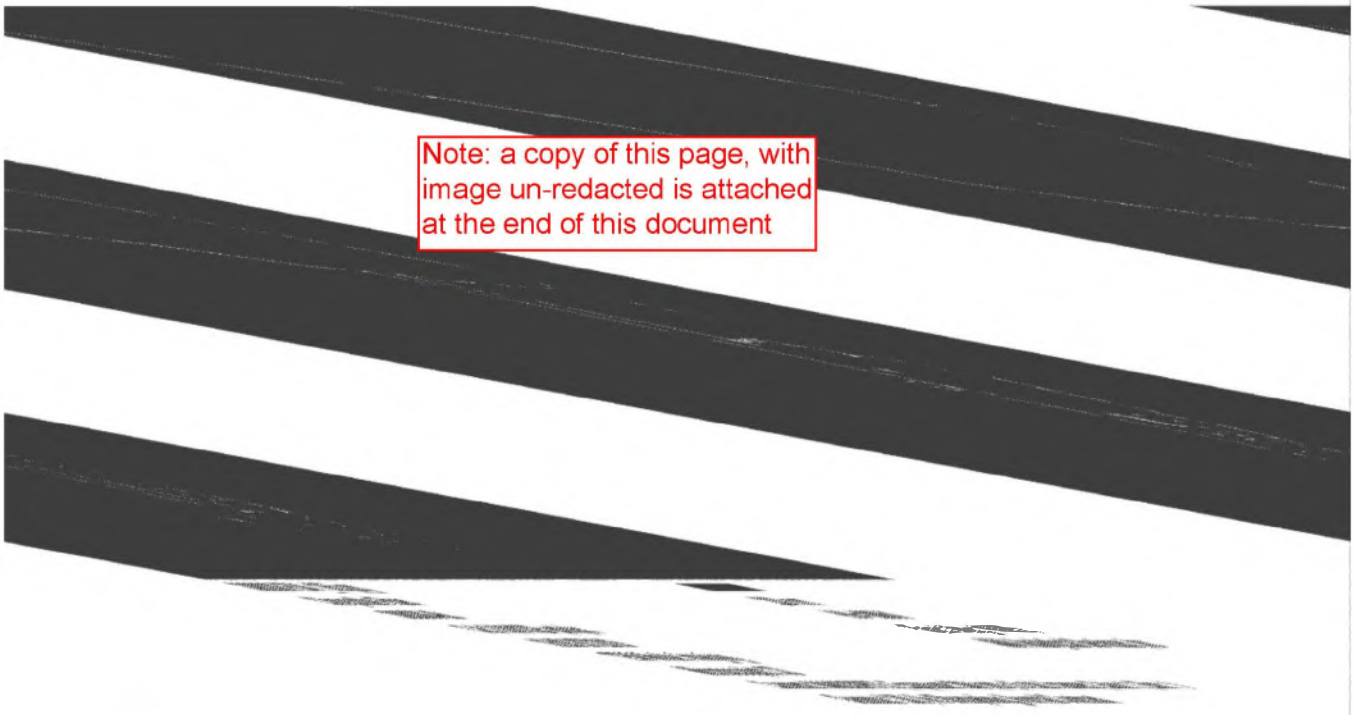
Chemistry from Chrysotile Structure pictured above



308006-6B, Chrysotile Structure 2



Note: a copy of this page, with image un-redacted is attached at the end of this document

Re: FDA Office of Cosmetics & Colors
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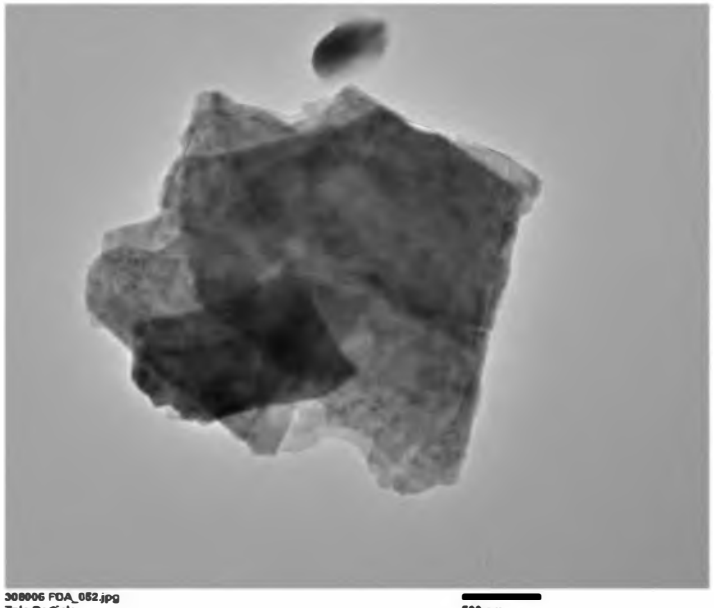
Diffraction Pattern from Chrysotile Structure pictured above

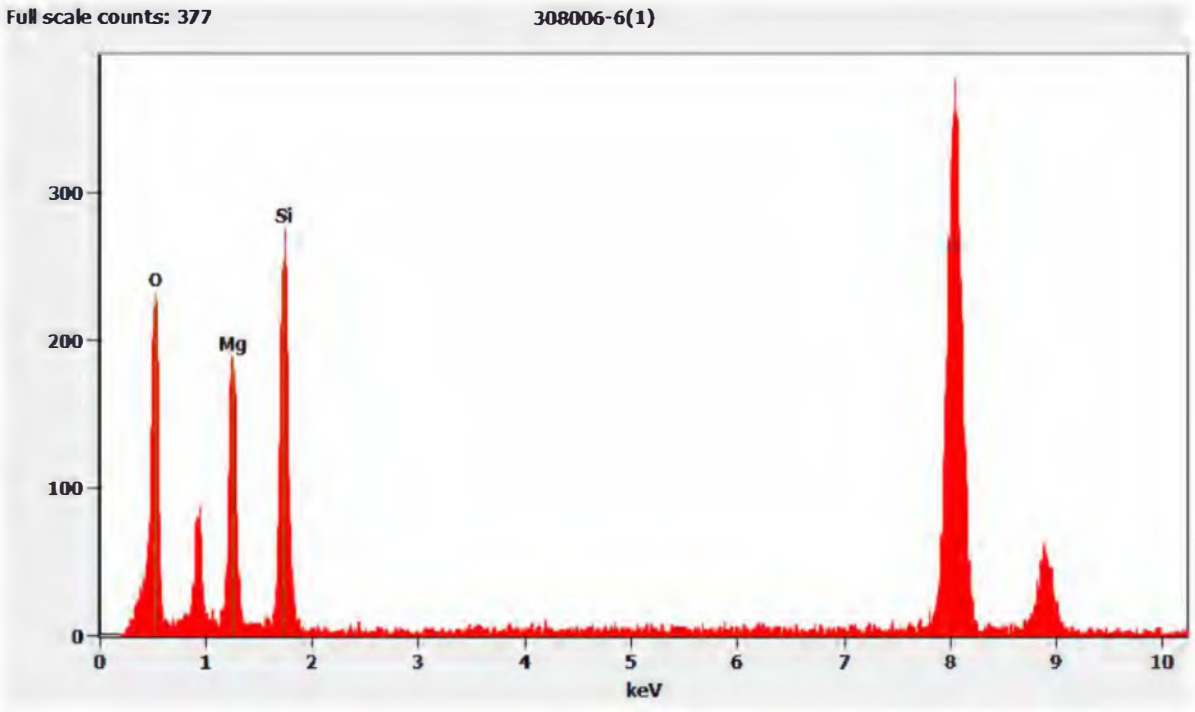
308006-6, Talc Particle



Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

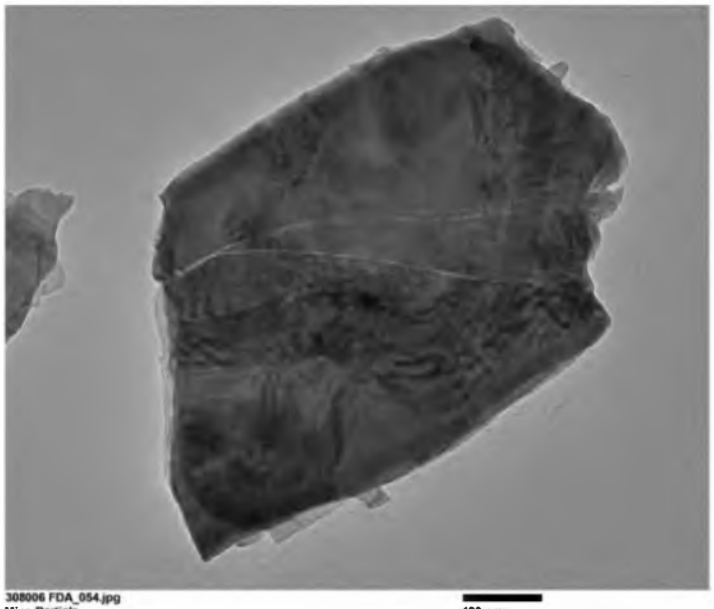
Hexagonal Diffraction Pattern from Talc Particle pictured above

Chemistry from Talc Particle pictured above



Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

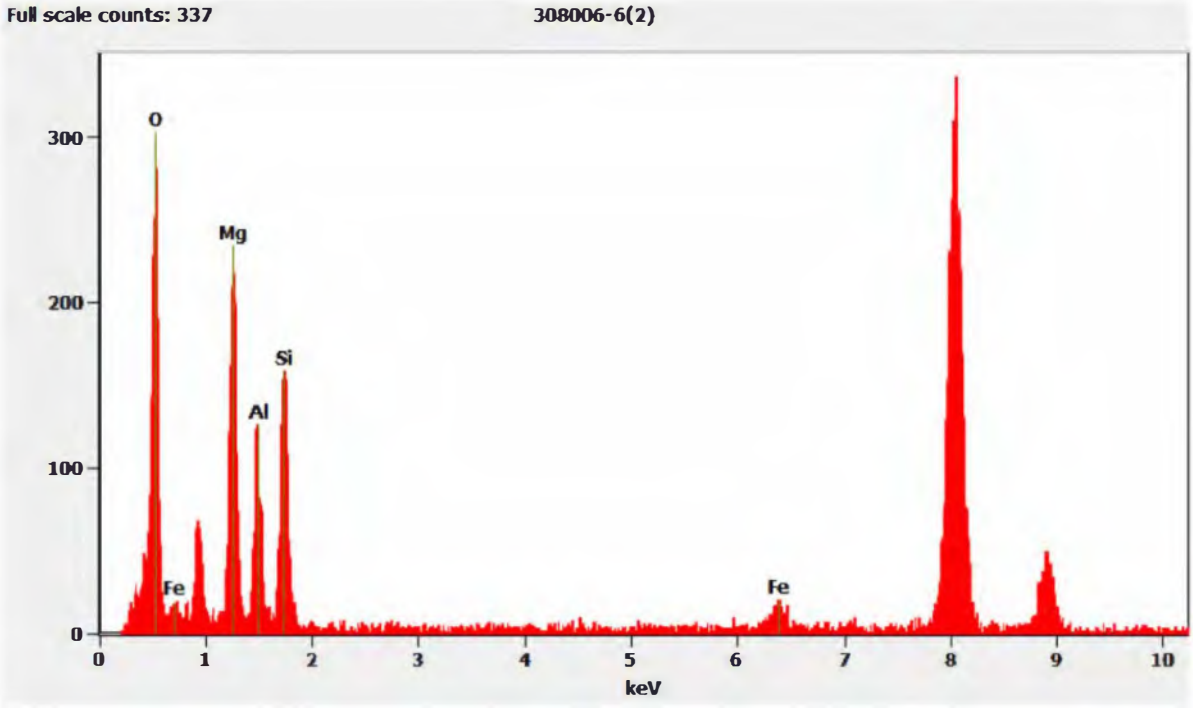
306008-6, Mica Particle



Diffraction Pattern from Mica Particle pictured above

Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

Chemistry from Mica Particle pictured above

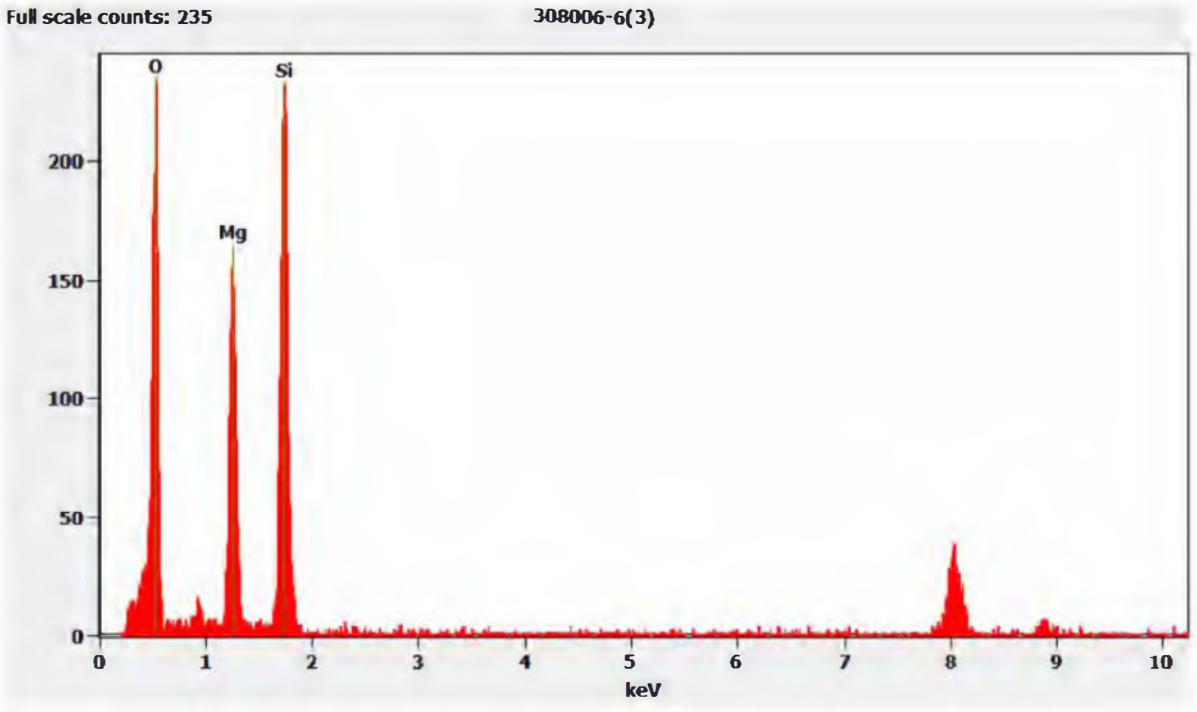


308006-6, Talc Fiber

Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

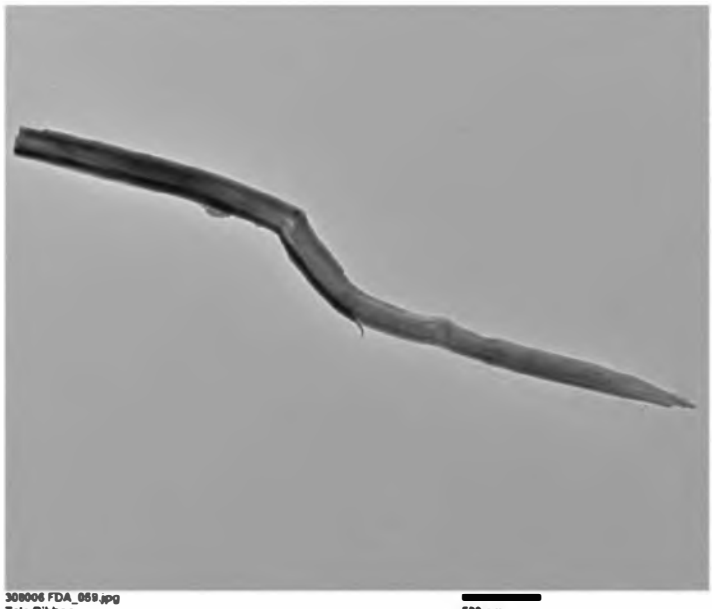
Diffraction Pattern from Talc Fiber pictured above

Chemistry from Talc Fiber pictured above



Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

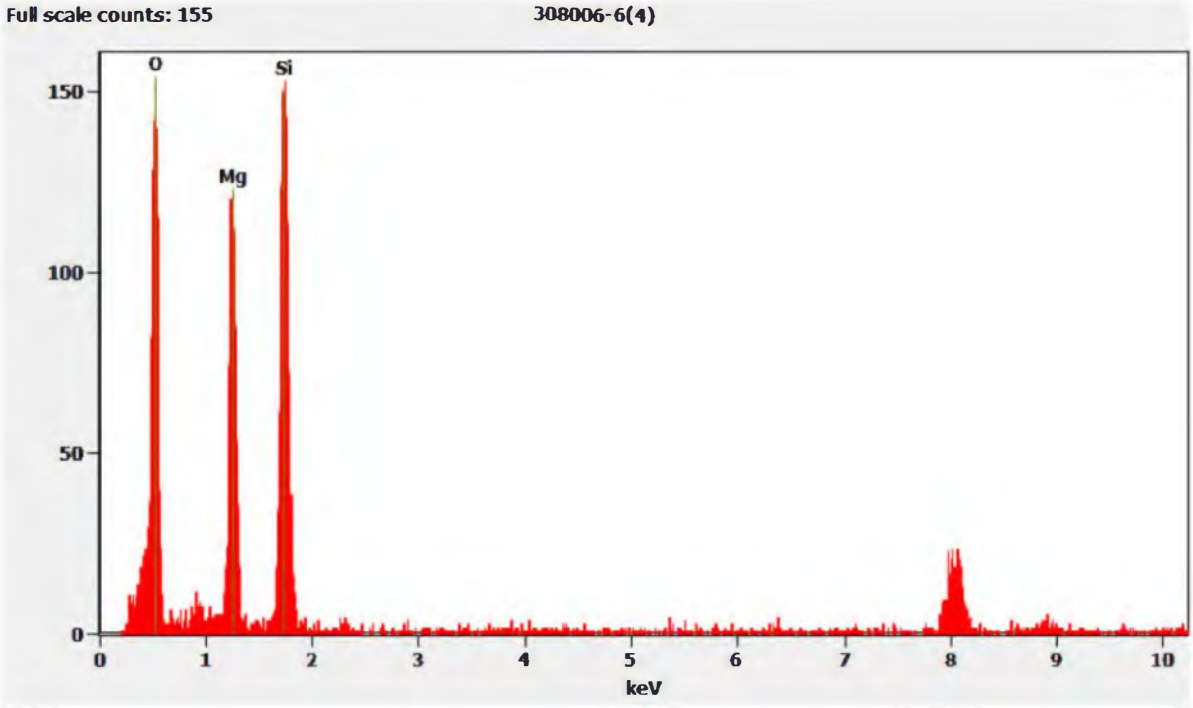
308006-6, Talc Ribbon



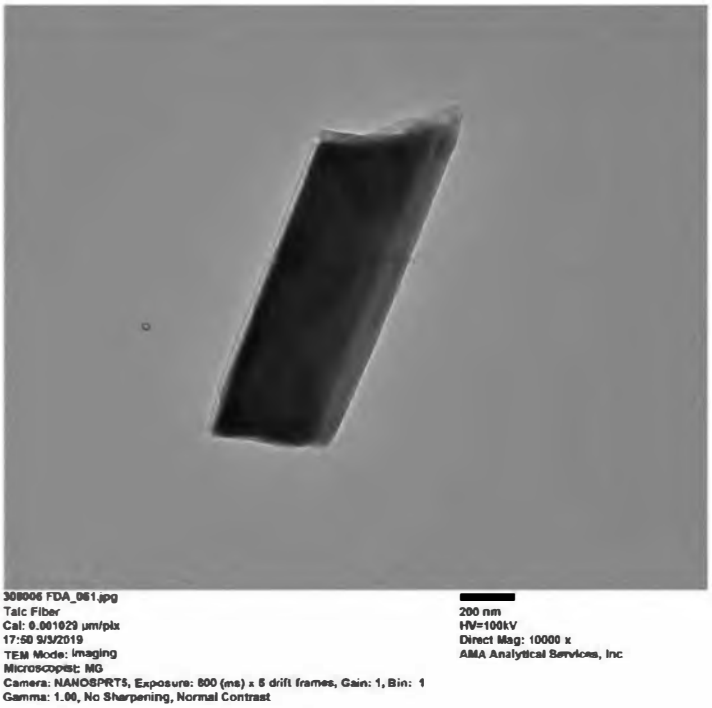
Diffraction Pattern from Talc Ribbon pictured above

Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

Chemistry from Talc Ribbon pictured above



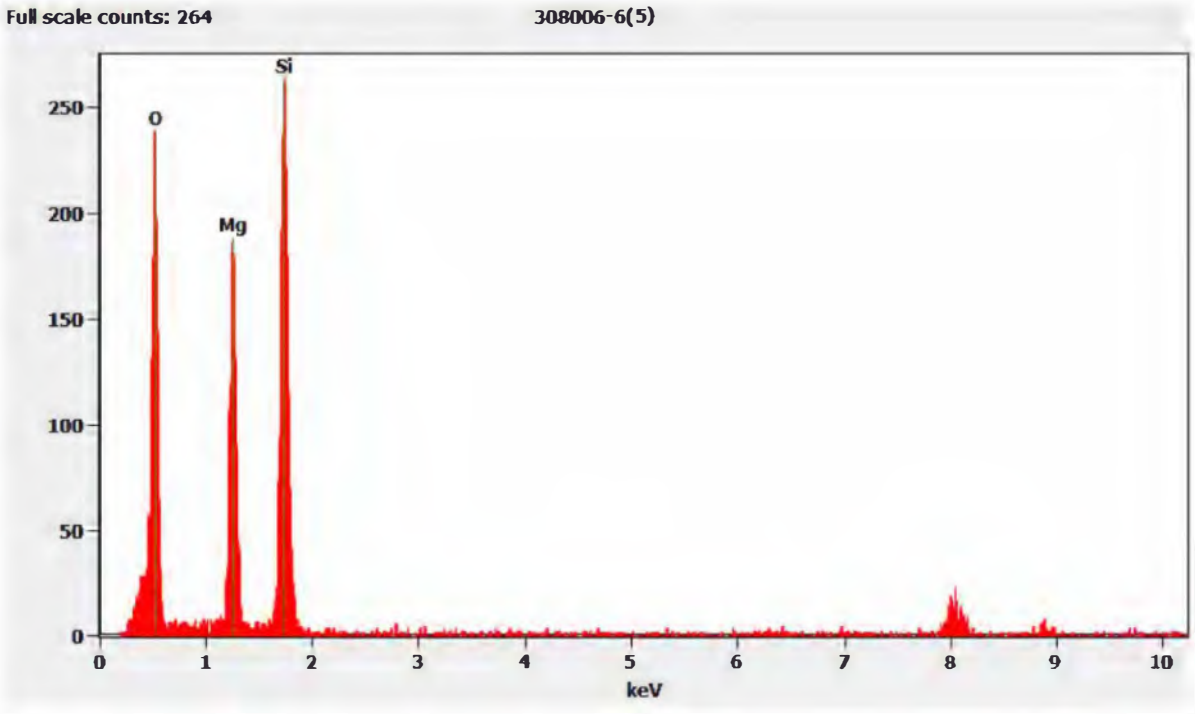
308006-6, Talc Fiber



Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

Diffraction Pattern from Talc Fiber pictured above

Chemistry from Talc Fiber pictured above



Re: FDA Office of Cosmetics & Colors
COC 308006-6, 6A,6B/D58, Revised 10/11/2019 (Revision #2)

QC Discussion:
During preparation, three blank control samples and one reference control sample were prepared. These samples were prepared alongside the customer samples. The blank samples were prepared using Sigma-Aldrich Talc Powder, <10 micron, and was analyzed by [redacted] on September 18, 2019. No asbestos was detected on the blank samples. The reference sample was made from the same Sigma-Aldrich talc powder spiked with 10% Chrysotile. The reference sample was analyzed by [redacted] on September 18, 2019 and found to be within acceptable limits. Additionally, filter blanks were prepared with each batch of carbon coated filters. Filter blank number EB-54155 was associated with the carbon coating for samples 308006-6, 6A, 6B/D-58. No asbestos was detected on the filter blank sample.

Our laboratory information management system (LIMS) randomly selected samples 308006-2/D-54 and 308006-15/D-67 for additional replicate QC analysis. Separate preparations were made for PLM and TEM analysis. The replicate QC analysis was performed by [redacted] on September 13, 2019, 2019 for PLM analysis and by [redacted] on September 18, 2019 for TEM analysis. The QC results matched the original analysis.

Attachments:
The following items are attached to this case narrative for your reference:

1) Sample Log-In Sheet
2) Daily PLM Scope Calibration Log
3) Refractive Index Oil Calibration Log
4) Daily TEM Scope Calibration Log
5) QC Results Summary
6) Replicate & Duplicate QC Chart for (b) (6) [redacted] for samples analyzed between 1/1/2019 and 9/18/2019
7) Replicate & Duplicate QC Chart for (b) (6) [redacted] for samples analyzed between 1/1/2019 and 9/18/2019
8) Replicate & Duplicate QC Chart for (b) (6) [redacted] for samples analyzed between 1/1/2018 and 9/18/2019
9) Raw Data Sheets
    a. Gravimetric Data
    b. Filtration Worksheets
    c. PLM Analysis
    d. TEM Analysis
    e. QC Samples

I certify that all information contained in this report pertaining to laboratory events, procedures, and protocols is true and accurately describes the handling of this project by AMA Analytical Services, Inc. and its personnel.

10/11/2019
Andreas Saldivar — Date
Laboratory Director

Login Sheet

Client:	US Food & Drug Adm n s ra on	**Job Name:**	Task 3 - Anay s s of Offc a Samp es	**Chain of Custody:**	308006
Date Submitted:	07/24/2019	**Job Location:**	4 h Group - 15 Samp es	**PO Number:**	SF225201810337P
Due Date:	09/13/2019 5:00 pm	**Job Number:**	CLIN 0001		

AMA Sample Number	Client Sample Number	Analysis Type(s) and Sample Type(s)
(b) (4)	(b)	(b) (4)

AMA Sample Number	Client Sample Number	Analysis Type(s) and Sample Type(s)
308006-6	D-58	PLM: ELAP 198.6 NOB TEM: Bulk Upgrade
308006-6A	D-58	PLM: ELAP 198.6 NOB TEM: Bulk Upgrade
308006-6B	D-58	PLM: ELAP 198.6 NOB TEM: Bulk Upgrade
308006-6C	D-58	PLM: ELAP 198.6 NOB TEM: Bulk Upgrade
(b) (4)	[redacted]	[redacted]

AMA Sample Number	Client Sample Number	Analysis Type(s) and Sample Type(s)
(b) (4)		

Special Instructions:
Use FDA Protocols. Samples are in Asbestos Sample Lock Box (See (b) (6) for Key). ALL PLM & TEM Analysts: Please record the date & amount of time spent analyzing each sample in the comments section of the bench sheet. Please save all pictures, graphs, etc. to L:\Case Narratives\FDA Project\308006

9/30/2019 by (b) (6): Client requested that we analyze a 4th aliquot for sample 308006 6/D58; this was added as 308006 6C
10/1/2019 by (b) (6): Client requested that we cancel their request to analyze 308006 6C, Preparation was mostly complete by the time we received the cancellation notice, but no analysis was performed.

Daily Calibrations for PLM Scope # 2 Date: 9/12/19
Analyst Initials: (b) (6)

Cleaning:
Oculars ✓ Objectives ✓ Field Lens ✓ Mechanical Stage ✓ Main Body ✓
Alignment:
✓ Kohler Illumination (or as close to it as scope allows)
✓ Polarizer & Analyzer at 90 degrees to one another
✓ Polarizer & Analyzer aligned with reticule cross hairs
✓ Axis of rotation of stage centered in field of view
✓ Central stop of the D.S. objective aligned with condenser aperture
Refractive Index Colors of Permanent 1,680 Amosite:
440 Parallel wavelength 660 Perpendicular wavelength

Comments/other procedures performed:

Daily Calibrations for PLM Scope # 2 Date: 9/13/19
Analyst Initials: [redacted]

Cleaning:
Oculars ✓ Objectives ✓ Field Lens ✓ Mechanical Stage ✓ Main Body ✓
Alignment:
✓ Kohler Illumination (or as close to it as scope allows)
✓ Polarizer & Analyzer at 90 degrees to one another
✓ Polarizer & Analyzer aligned with reticule cross hairs
✓ Axis of rotation of stage centered in field of view
✓ Central stop of the D.S. objective aligned with condenser aperture
Refractive Index Colors of Permanent 1,680 Amosite:
440 Parallel wavelength 660 Perpendicular wavelength

Comments/other procedures performed:

Daily Calibrations for PLM Scope # 2 Date: 9/16/19
Analyst Initials: (b) (6)

Cleaning:
Oculars ✓ Objectives ✓ Field Lens ✓ Mechanical Stage ✓ Main Body ✓
Alignment:
✓ Kohler Illumination (or as close to it as scope allows)
✓ Polarizer & Analyzer at 90 degrees to one another
✓ Polarizer & Analyzer aligned with reticule cross hairs
✓ Axis of rotation of stage centered in field of view
✓ Central stop of the D.S. objective aligned with condenser aperture
Refractive Index Colors of Permanent 1,680 Amosite:
440 Parallel wavelength 660 Perpendicular wavelength

Comments/other procedures performed:

REFRACTIVE INDEX OIL CALIBRATION

DATE	ANALYST	RI OIL	BOTTLE ID	LAB ID	RI BEAD	TEMP (Deg. C)	Dispersion Staining Color/ Becke Line Observations	Matching Wavelength	Measured R.I.
9-3-19	(b) (6)	1.550	1.550X3		1.55	25°	Red, Blue, Magenta	580	1.5505
〃		1.680	1.680X3		1.68	〃	Blue, Green	620	1.6816
〃		1.605	1.605X3		1.60	〃	Blue, Light Green	660	1.6041
〃		1.625	1.625X3		1.62	〃	Blue, Magenta	560	—
〃		1.700	1.700X3		1.70	〃	Blue, Green, Magenta	580	—

Revision 2, February 19, 2007

DAILY TEM CALIBRATION LOG

Every analyst should confirm alignment prior to analyzing samples.
X-ray analyzer must be calibrated prior to each day's use.
Dewar for x-ray detector is to be filled each Tuesday and Friday.

Note: Please enter code letters in Type Column.
Type of Analysis:

Routine Analysis	A
Quality Control	QC
Training	T
Research	R
Other (Explain)	O

TRANSMISSION ELECTRON MICROSCOPE								X-RAY ANALYZER	
DATE	NAME	SYSTEM/ ALIGN. CHECK	ACTUAL "BEAM TIME" USED ON	OFF	TOTAL MINUTES	TOTAL # SAMPLES	TYPE	EDXA CAL. (AL/CU)	DEWAR LN2 (INIT)
9/3/19	(b) (6)	OK							
9/4/19	(b) (6)	OK	Filament Changed						
9/5/19	(b) (6)	OK						GOK	
9/6/19	(b) (6)	OK	0900			12	A		
9/7/19	(b) (6)	OK	1230			17	A		
9/8/19	(b) (6)	OK							
9/9/19	(b) (6)	OK							
9/10/19	(b) (6)	OK							
9/11/19	(b) (6)	OK							
9/12/19	(b) (6)	OK							

Version 2-1:9/90

Revision 0, Issued Oct. 2004 BB

Chain Of Custody #308006

+ Add CoC

General | Samples | Documents | QC Results

QC Samples

Date Analyzed	Sample Number	Original PLM Analyst	Original PLM Result	PLM QC Result	PLM QC Analyst	PLM R Value	Original TEM Analyst	Original TEM Result	TEM QC Result	TEM QC Analyst	TEM R Value	Comments
09/09/2019	308006-16RQC	(b) (6)	0.00		SW	0.00	MG	0.00	0.00	CD	0.00	Analysis 9/18/19
09/09/2019	308006-17RQC		0.00		SW	0.00	MG	0.00	0.00	CD	0.00	Analysis: 9/18/19

Reference Samples

Sample Number	Tile #	Analyst	Asbestos Type	Percent Asbestos	Result	Created Date	Comments
Talc Ref	Talc Ref 10%	(b) (6)	Chrysotile	10.00	Pass	09/18/2019	

Blanks

Blank Number	Date		Asbestos Percentage	Asbestos Type	Comments
NB19-646	09/18/2019	(b) (6)	0.0		
NB19-645	09/18/2019		0.0		
NB19-647	09/18/2019		0.0		

PLM Error(s)

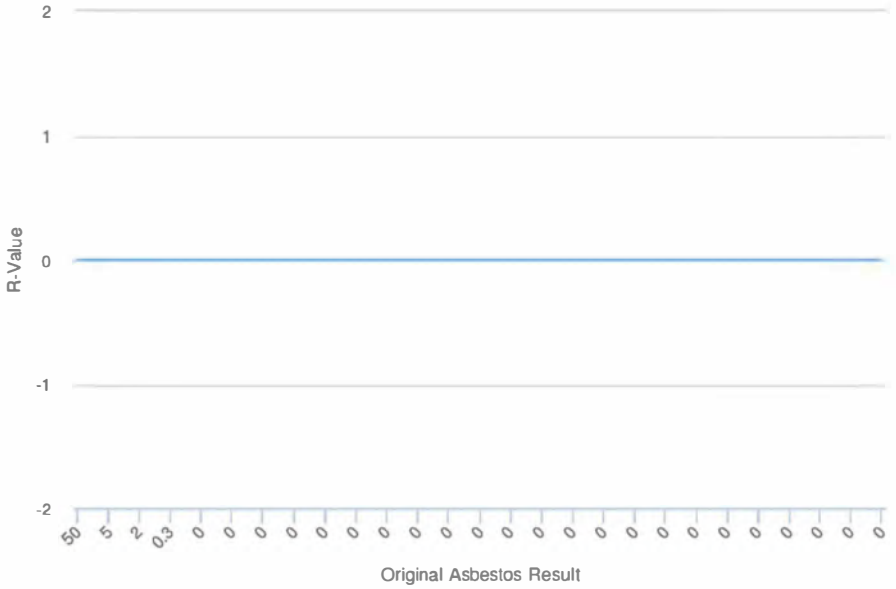
No Results

TEM NOB Error(s)

No Results

PLM QC Chart

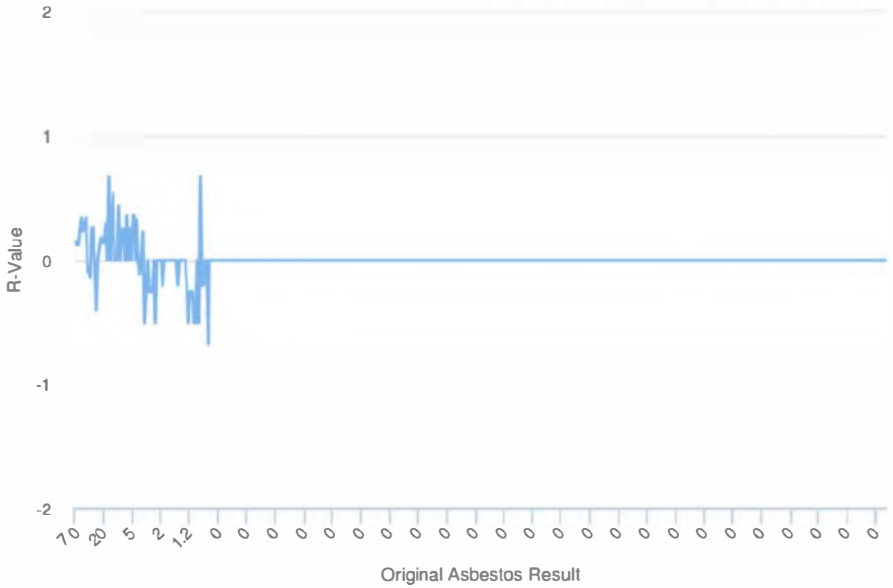
QC Type: Duplicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Original Result] - [QC Result] / [Average]

PLM QC Chart

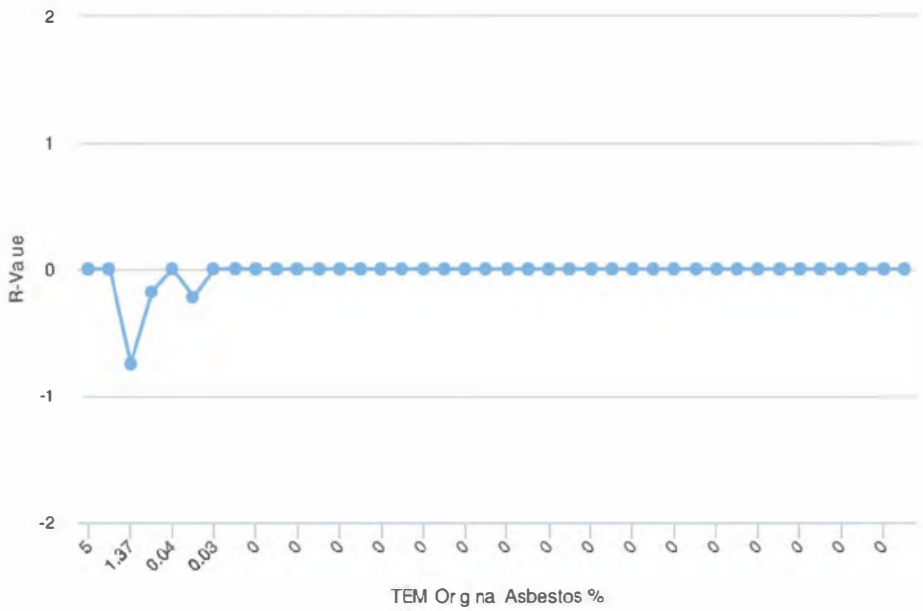
QC Type: Replicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Original Result] - [QC Result] / [Average]

NOB QC Chart

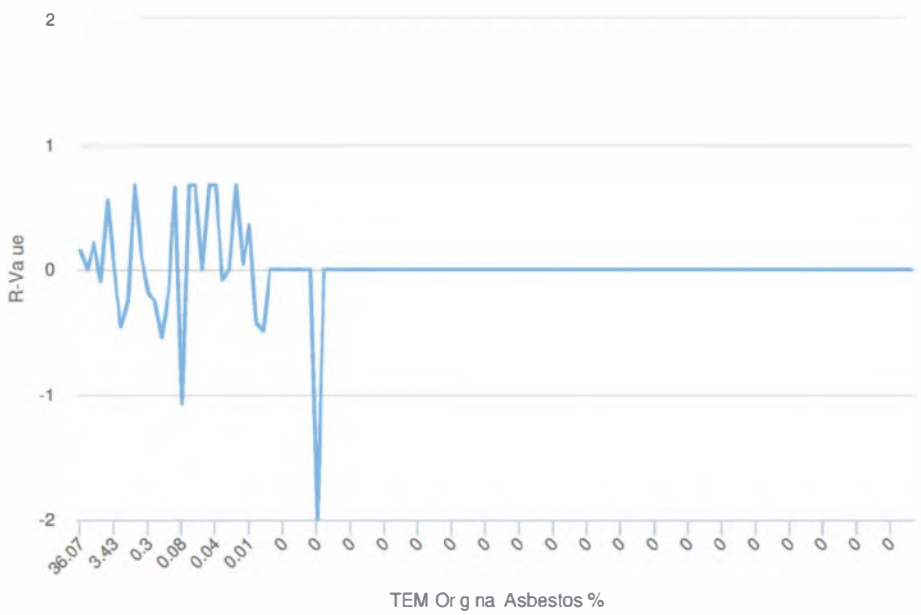
QC Type: Duplicate
Analyst (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Organa Result] - [QC Result] / [Average]

NOB QC Chart

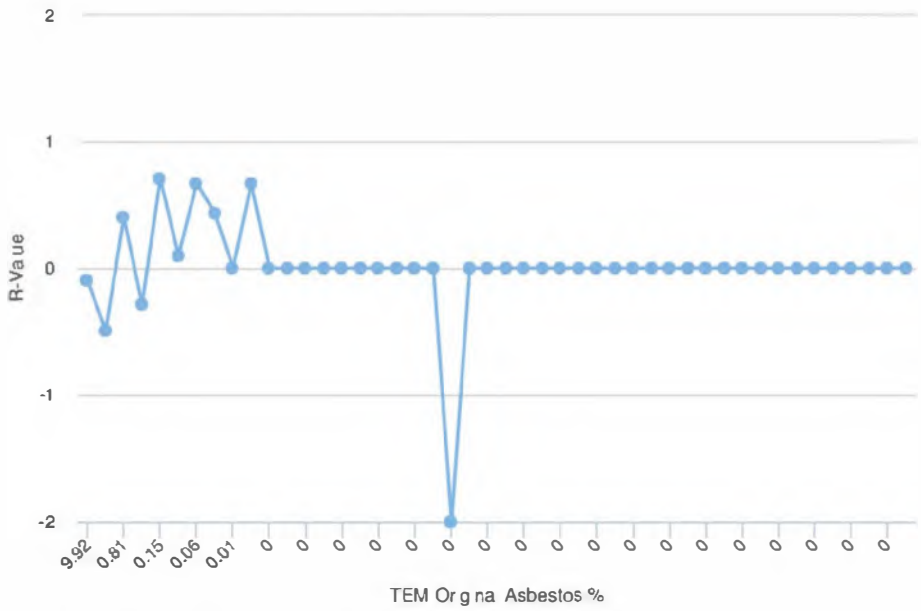
QC Type: Replicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Or g na Resu t] - [QC Resu t] / [Average]

NOB QC Chart

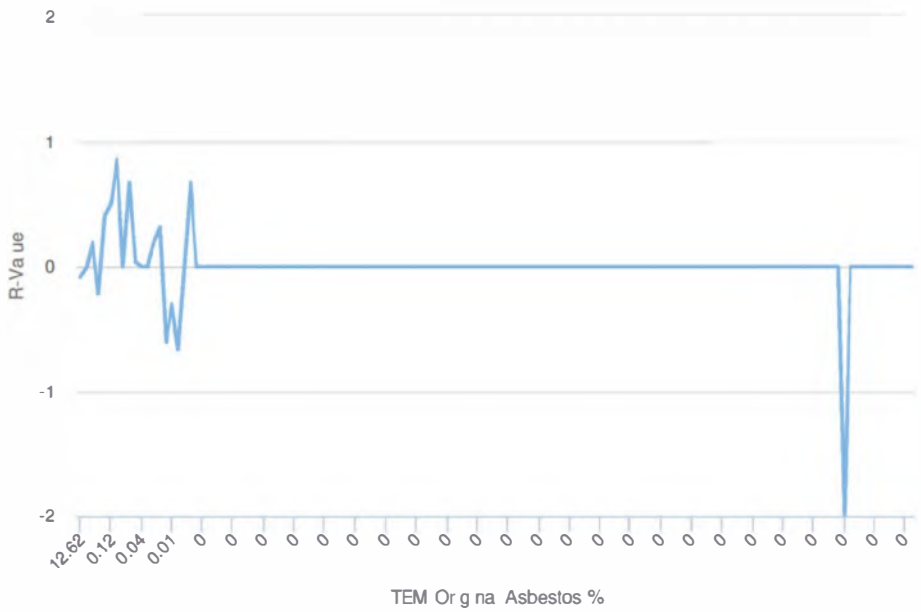
QC Type: Duplicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Organa Result] - [QC Result] / [Average]

NOB QC Chart

QC Type: Replicate
Analyst: (b) (6)
Dates Analyzed: *01/01/2019 - 09/18/2019*



R = [Organa Result] - [QC Result] / [Average]

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction and Filtration Bench Sheet for Modified ELAP 198.4

Page 1 of 3

TEM Preparations

COC #: 308006 Date: 8/13/2019 & 9/30/19

Client: USFDA Prep By: (b) (6)

Filter Type: 47 mm, 0.22 µm, MCE EFA: 1047 mm²

Filtered By: (b) (6) Lot #: R9CA03145 Date: See margin

AMA Sample ID	Gravimetric Reduction Weights: Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish	Filtration Weights: Mass (g) 100mL Jar w/ Lid	Mass (g) 100mL Jar w/ Lid & Sample Residue	Filtration Volumes: Initial Volume (mL)	Volume Filtered (mL)	Serial Dilution Initial Volume (mL) (if left blank, no serial dilution performed)	Serial Dilution Volume Filtered (mL) (if left blank, no serial dilution performed)	Serial Dilution Final Volume (mL) (if left blank, no serial dilution performed)
(b) (4)												
3080066	7.2520	7.6844	7.6833	6.0877	6.4902	19.6991	19.8935	100	.2			=
-6A	7.1474	7.6335	7.6324	6.0891	6.4791	19.7148	19.8949	100	.2			=
-6B	7.1687	7.6634	7.6622	6.0903	6.5285	19.8223	20.0189	100	.2			=
(b) (4)												=

8/19/2019

9/30/2019

Archive 1, Issued April 2019, ENH

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction and Filtration Bench Sheet for Modified ELAP 198.4

Page 2 of 3

TEM Preparations

COC #: 308006
Client: USFDA
Date: 8/13/19 & 9/30/19
Prep By: (b) (6) - 9/30 10/9/19

Filter Type: 47 mm, 0.22 µm, MCE EFA: 1047 mm²
Filtered By: (b) (6) Lot #: R9CA03145 ; Date: See margin 9/30 10/9/19

AMASampleID	Gravimetric Reduction Weights: Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish	Filtration Weights: Mass (g) 100mL Jar w/ Lid	Mass (g) 100mL Jar w/ Lid & Sample Residue	Filtration Volumes: Initial Volume (mL)	Volume Filtered (mL)	Serial Dilution Initial Volume (mL) (if left blank, no serial dilution performed)	Serial Dilution Volume Filtered (mL) (if left blank, no serial dilution performed)	Serial Dilution Final Volume (mL) (if left blank, no serial dilution performed)
(b) (4)	[redacted]											=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=
												=

8/30/2019

9/5/2019

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction and Filtration Bench Sheet for Modified ELAP 198.4

Page 3 of 3

TEM Preparations

COC #: 308006 Date: 8/13/19 - 9/30/19

Client: USFDA Prep By: (b) (6) - BKH 10/8/19

Filter Type: 47 mm, 0.22 µm, MCE EFA: 1047 mm²

Filtered By: (b) (6) Lot #: R9CA03145; Date: See margin - BKH 10/8/19

AMA Sample ID	Gravimetric Reduction Weights: Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Filtration Weights: Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish	Mass (g) 100mL Jar w/ Lid	Mass (g) 100mL Jar w/ Lid & Sample Residue	Filtration Volumes: Initial Volume (mL)	Volume Filtered (mL)	Serial Dilution Initial Volume (mL) (if left blank, no serial dilution performed)	Serial Dilution Volume Filtered (mL) (if left blank, no serial dilution performed)	Serial Dilution Final Volume (mL) (if left blank, no serial dilution performed)
(b) (4)	[redacted]											
NB19-645	7.2023	7.5055	7.5051	6.0260	6.2879	19.6999	19.8381	100	.2			=
NB19-646	7.1965	7.4457	7.4452	6.0241	6.2595	19.8538	19.9748	100	.2			=
NB19-647	7.1488	7.5222	7.5218	6.0210	6.3629	19.8468	20.0132	100	.2			=
RB								→	10%			=
9/30/19												=
308006-66	7.1397	7.5613	7.5601	61753	6.5876	20.8765	21.0656	100	.2			=
NB19-699	7.2313	7.5347	7.5335	6.2610	6.5578	19.8215	19.9581	100	.2			=
												=
												=

9/5/2019

10/8/19

Revision 1, Issued April 2018, DMH

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction Bench Sheet
Modified ELAP 198.6

PLM Preparations

Page 1 of 2

COC #: 308006 Date: 8/13/2019

Client: USFDA Prep By: (b) (6)

AMA Sample ID	Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish
(b) (4)					
308006-'6	7.1829	7.6946	7.6932	6.2233	6.6973
-6A	7.1097	7.5536	7.5526	6.1705	6.5757
-6B	7.2309	7.7182	7.7167	6.2312	6.6900
(b) (4)					

8/19/2019 = Post Silvation Dried Mass - BRH

8/30/2019 = Post Acid Wash Dried Mass - BRH

AMA Analytical Services, Inc.
Focused On Results.

Gravimetric Reduction Bench Sheet
Modified ELAP 198.6

PLM Preparations

Page 2 of 2

COC #: 308006 Date: 8/13/2019 -BMH 10/5/19

Client: USFDA Prep By: (b) (6)

AMA Sample ID	Mass (g) Vial	Mass (g) Vial & Sample	Mass (g) Post Ash Vial & Sample	Mass (g) Filter & Petri Dish	Mass (g) Post Acid Wash Filter & Petri Dish
(b) (4)					
NB19- 645	7.2023	7.5055	7.5051	6.0260	6.2879
NB19- 646	7.1965	7.4457	7.4452	6.0241	6.2595
NB19- 647	7.1488	7.5222	7.5218	6.0210	6.3629
RB					10 %

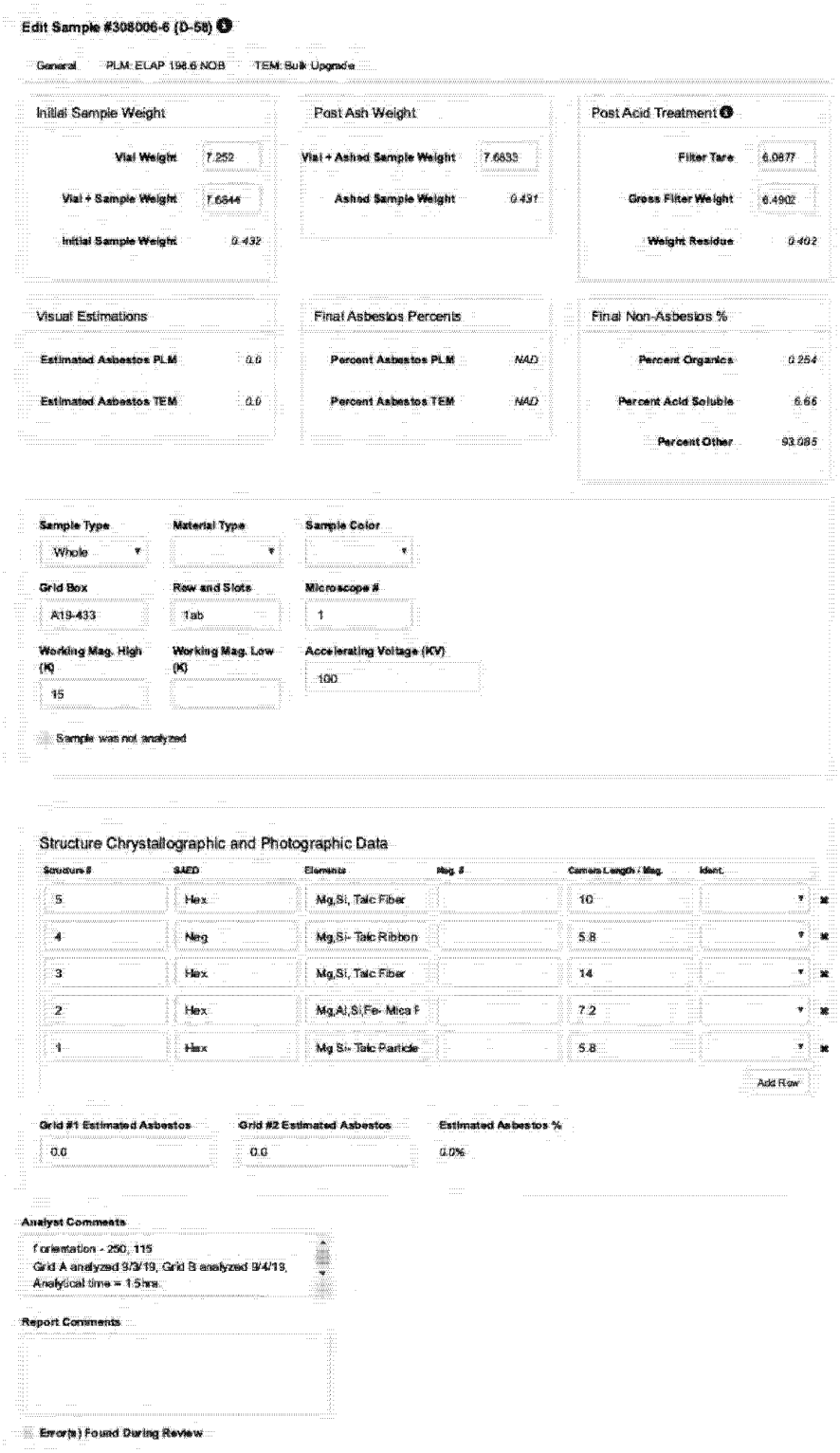
Per Elevation Data - BMH 10/5/19

8/30/2019

Per Elevation Data - BMH 10/5/19

9/5/2019


Edit Sample #308006-6 (D-56)
General
PLM: EAP 1668 NOB
TEM: Bulk Upgrade
Initial Sample Weight
Vial Weight
7.200
Vial + Sample Weight
7.6544
Initial Sample Weight
0.4544
Post Ash Weight
Vial + Ashed Sample Weight
7.6600
Ashed Sample Weight
0.4601
Post Acid Treatment
Filter Tare
0.2277
Gross Filter Weight
0.4560
Weight Residue
0.4601
Visual Estimations
Estimated Asbestos PLM
0.5
Final Asbestos Percents
Percent Asbestos PLM
NAD
Final Non-Asbestos %
Percent Organics
0.258
Percent Acid Soluble
2.56
Percent Other
97.045
Material Type
Powder
Color
White
Homogeneity
Homogeneous
Sample Type
Whole
Texture
Powdery
Sample was not analyzed
Fibrous
Scanning Negative Option
Stratified Point Count
400 Point Count
Micro Asbestos %
Chrysotile Percent
Amosite Percent
Crocidolite Percent
Other Asbestos Percent
Micro Non Asbestos %
Mineral Wool Percent
Fiberglass
Organic
Synthetic
Other
Micro Non Fibrous %
Particulate
100.0
Other Non-Fibrous
Macroscopic Comments
Temperature (°C)
25.0
Microscope #
2
Morphology
Color
Birefringence
Est Avg
Min 1
Min 2
Refractive Index 1
Refractive Index 2
Pleo
Birefringence
Refractive Index Of
Mark
Add Row
Size 1
Size 2
Size 3
Size 4
Size 5
Size 6
Size 7
Size 8
Mark
Fiber #
Add Row
Asbestos Points
Chrysotile Points
Pending
Trace Detected but no points
Amosite Points
Pending
Trace Detected but no points
Crocidolite Points
Pending
Trace Detected but no points
Other Asbestos Points
Pending
Trace Detected but no points
Total Asbestos Points
Pending
NAD
< 0.25%
Total Points
Pending
Non Asbestos Fiber Percentages
Mineral Wool + Fiberglass Combined
Organic
Synthetic
Other
Non Fibrous Percentages
Particulate
100.0
Other Non-Fibrous
Recommend TEM
Recommend Matrix Reduction
Microscopic Comments
Report Comments
Error(s) Found During Review



Edit Sample #308006-6 (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Bulk Upgrade

Initial Sample Weight

Vial Weight	7.252
Vial + Sample Weight	7.6844
Initial Sample Weight	0.432

Post Ash Weight

Vial + Ashed Sample Weight	7.6833
Ashed Sample Weight	0.431

Post Acid Treatment

Filter Tare	6.0877
Gross Filter Weight	6.4902
Weight Residue	0.402

Visual Estimations

Estimated Asbestos PLM	0.0
Estimated Asbestos TEM	0.0

Final Asbestos Percents

Percent Asbestos PLM	NAD
Percent Asbestos TEM	NAD

Final Non-Asbestos %

Percent Organics	0.254
Percent Acid Soluble	6.66
Percent Other	93.085

Sample Type	Material Type	Sample Color
Whole		

Grid Box	Raw and Slots	Microscope #
A19-433	Tab	1

Working Mag. High (K)	Working Mag. Low (K)	Accelerating Voltage (KV)
15		100

Sample was not analyzed

Structure Chrystallographic and Photographic Data

Structure #	SAED	Elements	Reg. #	Camera Length / Mag.	Ident.
5	Hex	Mg,Si, Talc Fiber		10	
4	Neg	Mg,Si- Talc Ribbon		5.8	
3	Hex	Mg,Si, Talc Fiber		14	
2	Hex	Mg,Al,Si,Fe- Mica F		7.2	
1	Hex	Mg Si- Talc Particle		5.8	

Add Row

Grid #1 Estimated Asbestos	Grid #2 Estimated Asbestos	Estimated Asbestos %
0.0	0.0	0.0%

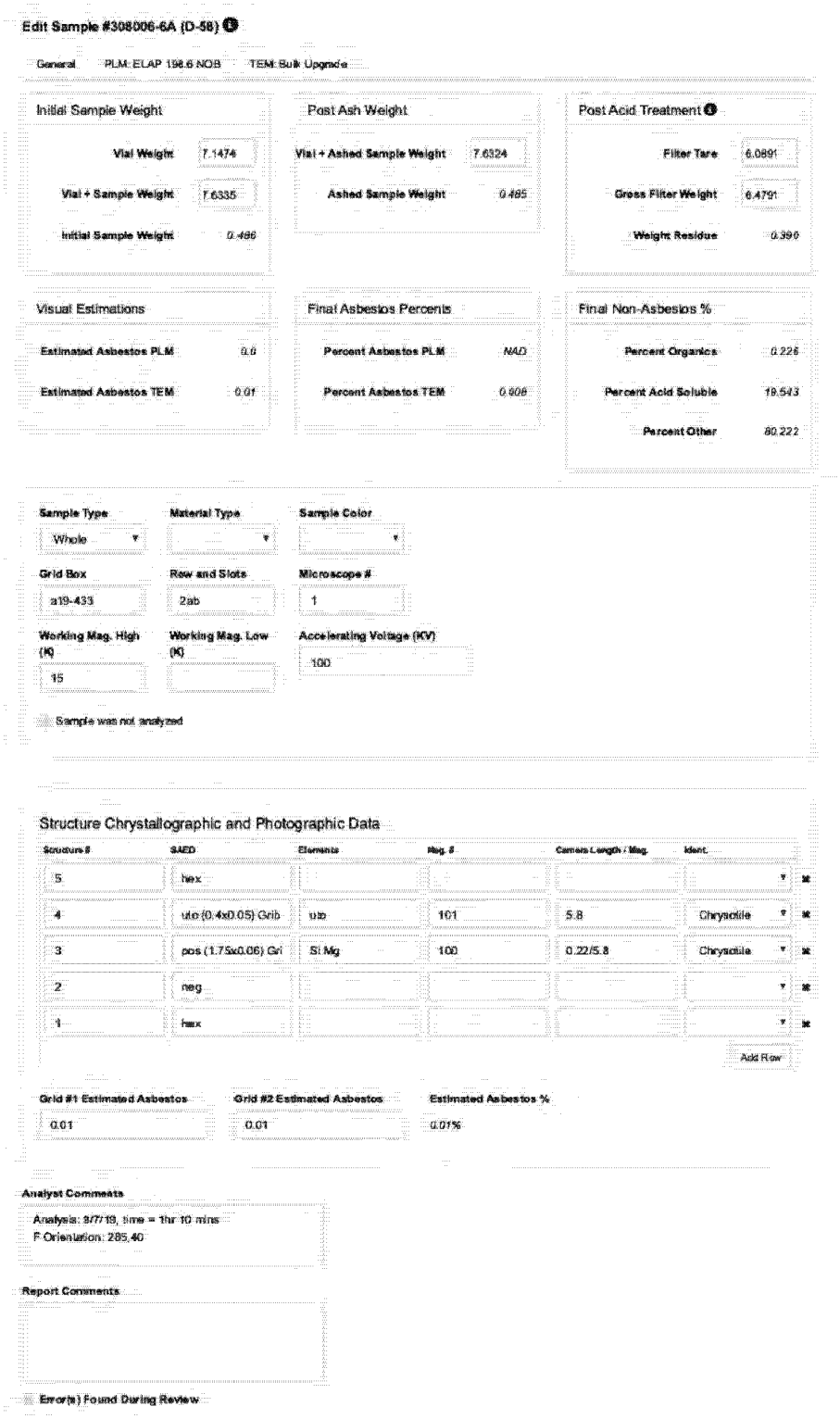
Analyst Comments

f orientation - 250, 115
Grid A analyzed 9/3/19, Grid B analyzed 9/4/19,
Analytical time = 1.5hrs

Report Comments

Error(s) Found During Review


Edit Sample #308006-6A (D-58)
General
PLM: ELAP 198.6 NOB
TEM: Bulk Upgrade
Initial Sample Weight
Vial Weight
7.1474
Vial + Sample Weight
7.6085
Initial Sample Weight
0.461
Post Ash Weight
Vial + Ashed Sample Weight
7.6524
Ashed Sample Weight
0.505
Post Acid Treatment
Filter Tare
0.0001
Gross Filter Weight
0.4791
Weight Residue
0.390
Visual Estimations
Estimated Asbestos PLM
0.5
Final Asbestos Percents
Percent Asbestos PLM
NAD
Final Non-Asbestos %
Percent Organics
0.226
Percent Acid Soluble
19.549
Percent Other
80.232
Material Type
Powder
Color
White
Homogeneity
Homogeneous
Sample Type
Whole
Texture
Powdery
Sample was not analyzed
Fibrous
Scanning Negative Option
Limited Point Count
400 Point Count
Micro Asbestos %
Chrysotile Percent
Amosite Percent
Crocidolite Percent
Other Asbestos Percent
Micro Non Asbestos %
Mineral Wool Percent
Fiberglass
Organic
Synthetic
Other
Micro Non Fibrous %
Particulate
100.0
Other Non-Fibrous
Macroscopic Comments
Temperature (°C)
26.6
Microscope #
2
Add Row
Asbestos Points
Chrysotile Points
Pending
Trace Detected but no points
Amosite Points
Pending
Trace Detected but no points
Crocidolite Points
Pending
Trace Detected but no points
Other Asbestos Points
Pending
Trace Detected but no points
Total Asbestos Points
Pending
NAD
< 0.25%
Total Points
Pending
Non Asbestos Fiber Percentages
Mineral Wool + Fiberglass Combined
Organic
Synthetic
Other
Non Fibrous Percentages
Particulate
100.0
Other Non-Fibrous
Recommend TEM
Recommend Matrix Reduction
Microscopic Comments
Report Comments
Error(s) Found During Review



Edit Sample #308006-6A (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Bulk Upgrade

Initial Sample Weight

Vial Weight	7.1474
Vial + Sample Weight	7.6335
Initial Sample Weight	0.486

Post Ash Weight

Vial + Ashed Sample Weight	7.6324
Ashed Sample Weight	0.485

Post Acid Treatment

Filter Tare	6.0891
Gross Filter Weight	6.4791
Weight Residue	0.390

Visual Estimations

Estimated Asbestos PLM	0.0
Estimated Asbestos TEM	0.01

Final Asbestos Percents

Percent Asbestos PLM	NAD
Percent Asbestos TEM	0.008

Final Non-Asbestos %

Percent Organics	0.226
Percent Acid Soluble	19.543
Percent Other	80.222

Sample Type	Material Type	Sample Color
Whole		

Grid Box	Row and Slots	Microscope #
a19-433	2ab	1

Working Mag. High (K)	Working Mag. Low (K)	Accelerating Voltage (KV)
15		100

Sample was not analyzed

Structure Chrystallographic and Photographic Data

Structure #	SAED	Elements	Mag. #	Camera Length / Mag.	Ident.
5	hex				
4	ute (0.4x0.05) Grid	uto	101	5.8	Chrysotile
3	pos (1.75x0.06) Gri	Si Mg	100	0.22/5.8	Chrysotile
2	neg				
1	hex				

Add Row

Grid #1 Estimated Asbestos	Grid #2 Estimated Asbestos	Estimated Asbestos %
0.01	0.01	0.01%

Analyst Comments

Analysis: 3/7/19, time = 1hr 10 mins
F Orientation: 285.40

Report Comments

Error(s) Found During Review

AMA Analytical Services, Inc.
Focused On Results.

Fiber Count Sheet for Transmission Electron Microscopy

Page 1 of 1

Date: 09/07/19 Client ID: D-58 Filter Size/Type/Porosity 47mm MCE 0.22 µm
Client: FDA COC #: 308006 AMA ID #: 308006-6A
Analyst: (b) (6) Working Mag: 15 K Accel Voltage: 100 kV Orientation of Letter F: F, R
Grid Box #: A19-433 Grid Acceptable: (Y) N Volume Filtered: 0.2 mL
Signed [redacted] Row #: 2 Grid: A / B / ___ Grid Openings to Observe: 20
Method: Mod. NY ELAP 198.4 (FDA Procedures)

Grid Opening # Location	Structure #	Length (in microns)	Width (in microns)	SAED # Rows	Mineral Type	Elements ID'd	Fiber Neg #	SAED Neg #	Magnification or Camera Length	
C2 B	1	1.75	0.06	pos	Chrysotile	Si, Mg	101	100	5.8 K	cm
C2 B	2	0.4	0.05	UTO	Chrysotile		101		5.8 K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
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									K	cm
									K	cm
									K	cm
									K	cm
									K	cm

Legend: NSD = No Structures Detected UTO = Unable to Obtain [ ] = 1st Grid [X] = 2nd Grid

# of Structures Counted: 2

	Mineral Type	= Total
Mineral Type:	Chrysotile	= 2
Mineral Type:		=
Mineral Type:		=
Mineral Type:		=

Total # of Grid Openings Observed: 20 = 0.280 mm²
Mean Grid Opening Area: 0.0140 mm²
EM Serial #: S/N 156120-35

Notes: Structure #2 determined to be Chrysotile asbestos based on morphology (tubular) Chemistry was UTO due to the size of the fibers surrounding particulate.

Revision 1, Issued April 2019 by QMR


Edit Sample #308006-01 (D-56)
General
PLM: ELAP 1966.NOB
TEM: Bulk Upgrade
Initial Sample Weight
Vial Weight
7.1697
Vial + Sample Weight
7.6634
Initial Sample Weight
0.495
Post Ash Weight
Vial + Ashed Sample Weight
7.6672
Ashed Sample Weight
0.494
Post Acid Treatment
Filter Tare
0.8880
Gross Filter Weight
0.8295
Weight Residue
0.438
Visual Estimations
Estimated Asbestos PLM
0.5
Final Asbestos Percents
Percent Asbestos PLM
NAD
Final Non-Asbestos %
Percent Organics
0.243
Percent Acid Soluble
11.778
Percent Other
88.07
Material Type
Powder
Color
White
Homogeneity
Homogeneous
Sample Type
Whole
Texture
Powdery
Sample was not analyzed
Fibrous
Scanning Negative Option
Stratified Point Count
400 Point Count
Micro Asbestos %
Chrysotile Percent
Amosite Percent
Crocidolite Percent
Other Asbestos Percent
Micro Non Asbestos %
Mineral Wool Percent
Fiberglass
Organic
Synthetic
Other
Micro Non Fibrous %
Particulate
100.0
Other Non-Fibrous
Macroscopic Comments
Temperature (°C)
25.0
Microscope #
2
Add Row
Asbestos Points
Chrysotile Points
Pending
Trace Detected but no points
Amosite Points
Pending
Trace Detected but no points
Crocidolite Points
Pending
Trace Detected but no points
Other Asbestos Points
Pending
Trace Detected but no points
Total Asbestos Points
Pending
NAD
< 0.25%
Total Points
Pending
Non Asbestos Fiber Percentages
Mineral Wool + Fiberglass Combined
Organic
Synthetic
Other
Non Fibrous Percentages
Particulate
100.0
Other Non-Fibrous
Recommend TEM
Recommend Matrix Reduction
Microscopic Comments
Report Comments
Error(s) Found During Review

Edit Sample #308006-6B (D-58)

General | PLM: ELAP 198.6 NOB | TEM: Sub Upgrade

Initial Sample Weight		Post Ash Weight		Post Acid Treatment	
Vial Weight	7.1687	Vial + Ashed Sample Weight	7.6622	Filter Tare	6.0903
Vial + Sample Weight	7.6634	Ashed Sample Weight	0.494	Gross Filter Weight	6.5285
Initial Sample Weight	0.495			Weight Residue	0.438

Visual Estimations		Final Asbestos Percents		Final Non-Asbestos %	
Estimated Asbestos PLM	0.0	Percent Asbestos PLM	NAD	Percent Organics	0.243
Estimated Asbestos TEM	0.01	Percent Asbestos TEM	0.009	Percent Acid Soluble	11.178
				Percent Other	88.57

Sample Type	Material Type	Sample Color
Whole		

Grid Box	Raw and Slots	Microscope #
A19-433	3ab	1

Working Mag. High (K)	Working Mag. Low (K)	Accelerating Voltage (KV)
15		100

Sample was not analyzed

Structure Chrystallographic and Photographic Data

Structure #	SAED	Elements	Mag. #	Camera Length / Mag.	Ident.
5	hex				
4	neg				
3	hex				
2	pos (1.0x0.05, Matr	Si Mg Al	104	0.22/10000	Chrysotile
1	pos (1.6x0.05, Clus	Si Mg	102	0.22/7800	Chrysotile

Add Row

Grid #1 Estimated Asbestos	Grid #2 Estimated Asbestos	Estimated Asbestos %
0.01	0.01	0.01%

Analyst Comments

Analysis: 09/07/2019, Time = 1hr 16 mins
F Orientation: 120,230
Al peak on the chrysotile matrix is likely from the

Report Comments

Error(s) Found During Review

AMA Analytical Services, Inc.
Focused On Results.

Fiber Count Sheet for Transmission Electron Microscopy

Page 1 of 1

Date: 09/07/19 Client ID: D-58 Filter Size/Type/Porosity 47mm MCE 0.22 µm

Client: FDA COC #: 308006 AMA ID #: 308006-68

Analyst: (b) (6) Working Mag: 15 K Accel Voltage: 100 kV Orientation of Letter F: 2, 4

Grid Box #: A19-433 Grid Acceptable: (Y) N Volume Filtered: 0.2 mL

Signed: Row #: 3 Grid: A / B / Grid Openings to Observe: 20

Method: Mod. NY ELAP 198.4 (FDA Procedures)

Grid Opening # Location	Structure #	Length (in microns)	Width (in microns)	SAED # Rows	Mineral Type	Elements ID'd	Fiber Neg #	SAED Neg #	Magnification or Camera Length	
F9 B	1a	1.6	0.05	pos	Chrysotile	Si, Mg	103	102	7.2 K	cm
F8 B	1b	1.1	0.05	pos	Chrysotile	Si, Mg	103	102	7.2 K	cm
F8 B	1c	0.75	0.04	pos	Chrysotile	Si, Mg	103	102	7.2 K	cm
E3 A	2	1.0	0.05	pos	Chrysotile	Si, Mg, Al	105	104	10 K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
									K	cm
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									K	cm
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									K	cm
									K	cm
									K	cm
									K	cm

Legend: NSD = No Structures Detected UTO = Unable to Obtain [ ] = 1st Grid [X] = 2nd Grid

Mineral Type: Chrysotile = Total 4 Total # of Grid Openings Observed: 20 = 0.280 mm² # of Structures Counted: 4

Mineral Type: = Mean Grid Opening Area: 0.0 140 mm² Notes: Al peak is likely

Mineral Type: = from the surrounding

Mineral Type: = EM Serial #: S/N 15612D-35 particulate on Structure #2

Revision 1, Issued April 2019 by DJH

Edit TEM NOB Blank Result

Blank Number

NB19-645

Analyst

(b) (6)

Date Analyzed

09-18-2019

Percent Asbestos

0.0

Asbestos Type

Comments

Save Changes

Edit TEM NOB Blank Result

Blank Number

NB19-646

Analyst

(b) (6)

Date Analyzed

09-18-2019

Percent Asbestos

0.0

Asbestos Type

Comments

Save Changes

Edit TEM NOB Blank Result

Blank Number

NB19-647

Analyst

(b) (6)

Date Analyzed

09-18-2019

Percent Asbestos

0.0

Asbestos Type

Comments

Save Changes

Edit NOB Reference Sample Result

Sample Number	Analyst	Reference Sample Tile #
Talc Ref	Christopher C	Talc Ref 10%

Reference Value	Asbestos Type	Lower Limit	Upper Limit
10	Chrysotile	5	25

Vial Weight	0.0	Post Acid Weight	1.0	Asbestos Type	Chrysotile
Vial and Sample Weight	1.0	Filter Tare	0.0	Estimated Asbestos	10.0
Vial and Ashed Sample Weight	1.0	Ashed Weight	1	Percent Asbestos	10
Initial Sample Weight	1	Residue Weight	1	Result	Pass

Comments

Save Changes

AMA Analytical Services, Inc.
Laboratory Blank Log

Blank ID #	Initials	Prep Date	Chain of Custody #	AMA or Client Sample Numbers	Analysis Date	Asbestos Conc.	Client Name	Archive Box #
EB- 54140	(b) (6)	8/30/19	[redacted]					
EB- 54141		8/30/19						
EB- 54142		8/30/19						
EB- 54143		"						
EB- 54144		"						
EB- 54145		8/31/19						
EB- 54146		"						
EB- 54147		"						
EB- 54148		"						
EB- 54149		9/1/19						
EB- 54150		9/3/19						
EB- 54151		"						
EB- 54152		"						
EB- 54153		"						
EB- 54154		"						
EB- 54155		"	308006	308006 (6, 6A, 6B	(b) (4)		USFDA	
EB- 54156		"	(b) (4)				USFDA	
EB- 54157		"					USFDA	
EB- 54158		9/4/19						
EB- 54159		"						
EB- 54160		"						
EB- 54161		"						

Ver: 1-3 (4/91)

Revision 0, Issued Oct 2004 AS 10/4/19

Edit Air Blank Result

Blank Number

54155

Analyst

(b) (6)

Date Analyzed

09-18-2019

Area Analyzed

0.07

Asbestos Structures

0

Asbestos Type

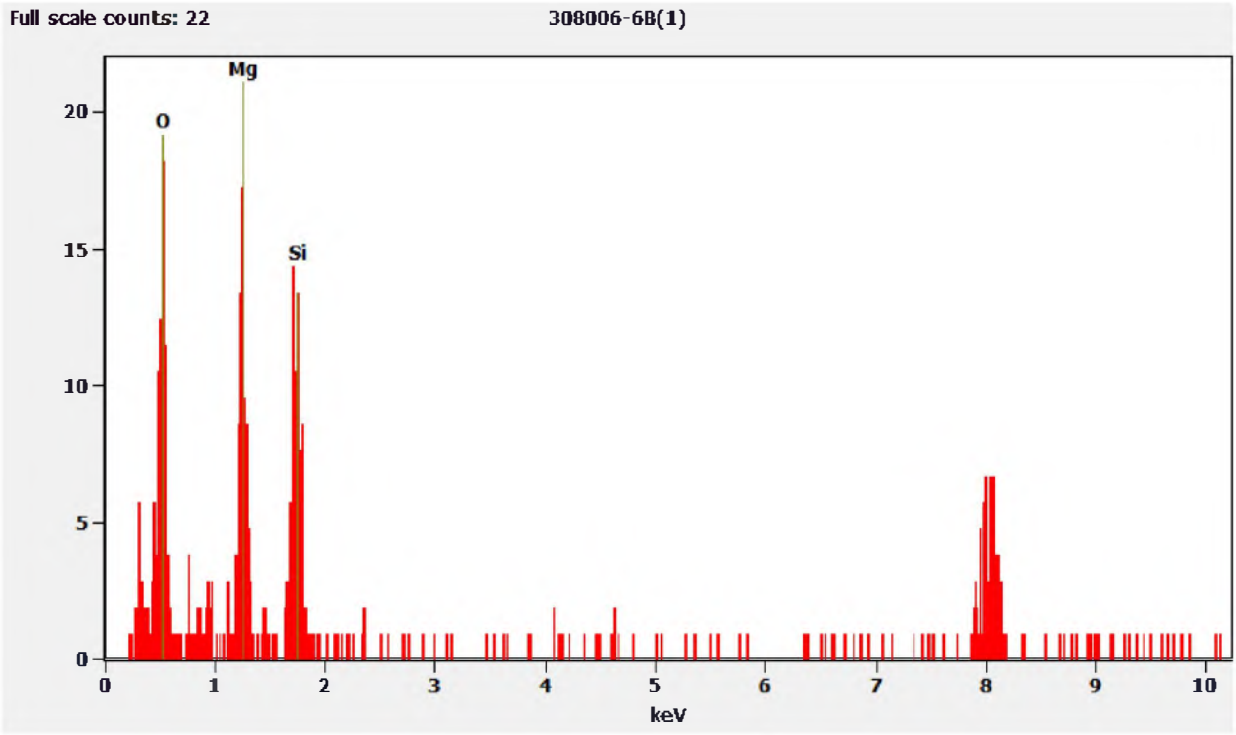
Result

< 14.286

Comments

Save Changes

Chemistry from Chrysotile Structure pictured above



308006-6B, Chrysotile Structure 2

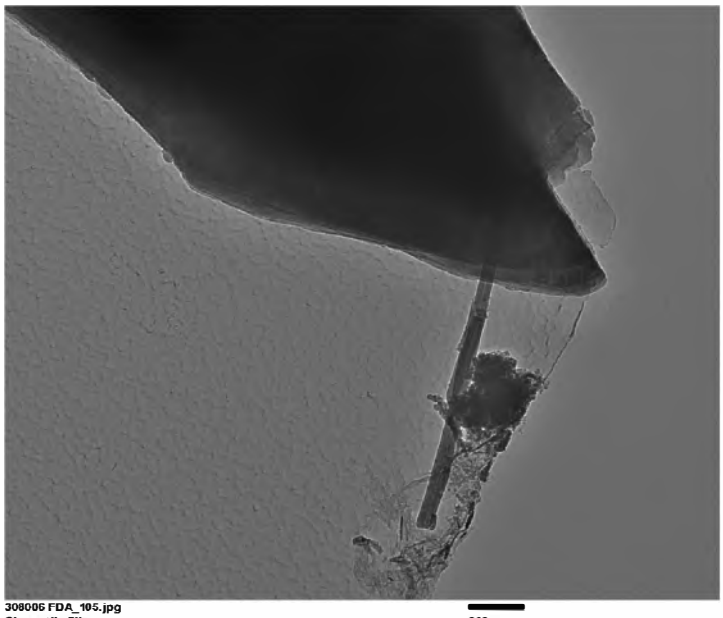


Exhibit L

Environmental Health Perspectives
Vol. 94, pp. 225–230, 1991

Amphibole Content of Cosmetic and Pharmaceutical Talcs

by A. M. Blount*

Pharmaceutical and cosmetic-grade talcs were examined for asbestiform amphibole content using a new density-optical method. Talcs under the Food and Drug Administration are not regulated as to asbestos content; however, all talcs were well below the level mandated by the Occupational Safety and Health Administration for industrial talcs. Only one was found to contain an amphibole particle size distribution typical of asbestos.

Introduction

In 1973 the Food and Drug Administration (FDA) proposed a regulation on the permissible asbestos content of talc (*1*). This regulation proposed to limit the amount of amphibole minerals to less than 0.1% and chrysotile to less than 0.01%. However, the optical microscopy method proposed was so complicated, lengthy, and subject to error that the proposed method was never finalized. Since then no final ruling has been issued.

The Occupational Safety and Health Administration, on the other hand, has been more rigorous and has instituted regulations despite the lack of methods to carry out the required measurements. One regulation, instituted in 1986, defines amphibole minerals as asbestos if the length to width ratio is 3:1 or greater. Because many nonfibrous cleavage fragments of amphibole minerals have a 3:1 aspect or greater and because there is no good evidence for adverse effects of these particles, a stay has been in affect on this part of the regulation (*2*). The second applicable regulation is the Hazard Communication Regulation (*3*), which applies to all chemicals used in the workplace. Specifically, it requires labeling of substances containing > 1% of a chemical hazardous to health and > 0.1% of a carcinogenic chemical.

Unfortunately, asbestos and amphiboles cannot be measured using currently developed methods to the level of 0.1% in the presence of talc. Some investigators have suggested that tremolite can be measured to that level by X-ray diffraction. But others have shown that the peak intensities vary between nonfibrous and fibrous tremolite (*4*) so that the 0.1% level of detection and measurement is doubtful except in cases where the sample has been spiked so that the exact nature of the tremolite is known. For anthophyllite there is little argument about the fact that detection cannot be made to 0.1%. However, the main problem with using X-ray diffraction for detection of amphibole minerals is that it gives no information about the shape of the particles, and shape is important in view of the uncertainity in the outcome of the asbestos regulation pertaining to nonfibrous amphiboles.

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The talcs that are pharmaceutical grade fall under the domain of the FDA and are therefore nonregulated in regard to fibrous mineral content. In the course of developing a technique to facilitate quantification of amphiboles in talc (*5*), pharmaceutical and high-grade talcs were examined. They were found to have very low amphibole content and, because of this, were extensively used in examining the lower limit of detection of the new method. The purpose of this paper is to describe the results of analyses for content and shape of amphibole mineral fragments in cosmetic and pharmaceutical talc powders of the United States.

Methods

The method proposed by the FDA in 1973 for analysis of talc was an optical procedure as described below (*1*):

> Weigh out 1 milligram of a representative portion of talc on each of two microscope slides. Mix the talc with a needle on one slide with a drop of 1.574 refractive index liquid, and then the other with 1.590 liquid, and place on each a square or rectangular cover glass sufficiently large so that the liquid will not run out from the edge (ca. 18 mm square) and will provide a uniform particle distribution. Fibers counted by this method should meet the following criteria: (i) Length to width ratio of 3 or greater (ii) length of 5 μm or greater (iii) width of 5 μm or less. Count and record the number of asbestos fibers in each 1 milligram as determined from a scan of both slides with a polarizing microscope at a magnification of approximately 400×. In the 1.574 refractive index liquid, chrysotile fibers with indices less than 1.574 in both extinction positions may be present: in the 1.590 refractive index liquid, the other five amphibole types of asbestos fibers with indices exceeding 1.590 in both extinction positions may be present. Check the extinction and sign of elongation for tentative identification. For specific identification of asbestos fibers, make additional mounts in appropriate refractive index liquids, and refer to the optical crystallographic data in the table. A count of not more than 1000 amphibole types of asbestos and not more than 100 chrysotile asbestos fibers per milligram-slide constitutes the maximum limit for the presence of these asbestos fibers in talc. These limits assure a purity of at least 99.9 percent free of amphibole types of asbestos fibers and at least 99.99 percent free of chrysotile asbestos fibers.

The problem with the proposed method is that talc flakes are often oriented vertically or at a sufficient angle that they appear to be needles and thus must be tested for refractive index (Fig. 1). A typical number of such particles is five per field of view. This

FIGURE 1. Talc flakes in 1.584 refractive index liquid. Note that there are particles in this field that have aspect ratios greater than 3:1. Width of view 0.13 mm.

means that some 20,000 particles would need to be examined in a typical case. In additon, chlorite is often present and when on edge must be examined in two extinction positions. This is clearly beyond what could be expected of any sane microscopist for a routine analysis. Since no other procedure has been developed as an alternative, a compromise has been to count 100 fields of view (FOV). In this way one need only examine about 500 particles in detail.

Because 500 particles is still a lengthy process, a more rapid and equally accurate method has been developed based on concentrating the amphibole particles by density difference. Figure 2 illustrates that there is a distinct break in density ranges between talcs and amphiboles. A heavy liquid of intermediate density is used, either Klein's (cadmium borotungstate) or Clerici's (thallium formate-malonate) solution. Experimentation showed that a heavy liquid of density 2.810 gives good separation even though values given in the literature and shown in Figure 2 would suggest that the density should be slightly higher. Because the density difference between particles and liquid is small, to get separation in a reasonable length of time a microcentrifuge is used with tubes containing 1.5 mL liquid. The height of the liquid column is, in this case, about 10 mm.

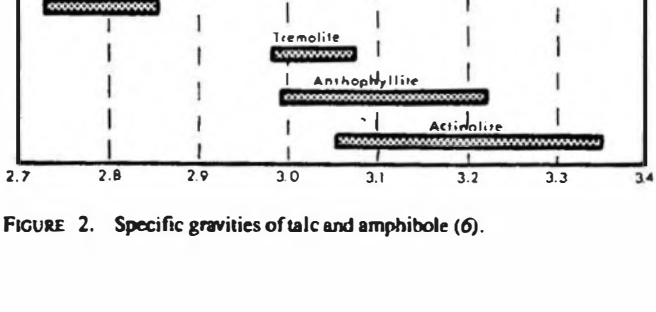

FIGURE 2. Specific gravities of talc and amphibole (6).

The general procedure involves weighing about 60 mg sample into a microcentrifuge tube and adding heavy liquid of density 2.810. After these are mixed, the tube with sample is placed in a vacuum for 3 min to remove the small bubbles adhering to the particles. After centrifuging the sample for 10 min at 7000 rpm, the heavy particles are removed from the bottom of the tube with a micropipette.

The counting of particles can be done either on a membrane filter (Nuclepore, 1.0 μm pore size) which has been placed on a microscope slide or as particles directly on the glass slide. In the first case, the heavy liquid with sample is forced through a membrane filter followed by distilled water to clean out the heavy liquid. The filter is then placed on a glass slide while wet. When dry, 1.584 refractive index liquid is placed on the filter followed by a cover glass. The photographs shown in this paper are of particles on filters.

The second case, particles directly on the microscope slide, requires transferring the heavy particles and some of the heavy liquid to a second centrifuge tube. Distilled water is added and the sample centrifuged. The liquid is pipetted off and more distilled water added. This is repeated several times to clean out the heavy liquid. Finally, the particles with several drops of water are transferred to a glass microscope slide. The advantage of this procedure is that any refractive index liquid can be used, whereas, in the former case, the refractive index is constrained by having to match the index of the membrane filter (either 1.584 or 1.625). The 1.584 value is good for analyzing amphiboles in talc, but the centrifuge method described has application to other mineral combinations, such as talc-quartz. With other combinations, refractive indices other than the two exhibited by the membrane filter may be more appropriate.

The particles are counted in 20 FOV. Being concentrated from 60 mg or more of sample, one will see more amphiboles than in 100 FOV using the old method. The number of amphibole particles per milligram (ppmg) is calculated:

$$\text{ppmg} = \text{amphibole particles/mg} = \frac{(\text{number of amphibole counted/number FOV counted}) \times \text{total number FOV}}{(\text{efficiency}) \times (\text{number of mg of sample})}$$

Efficiency of the spin-down is determined experimentally. For more details of the method see Blount (5).

Figure 3 illustrates the results obtained when testing the method using known mixtures. Because it is difficult to measure and mix in very small weights of amphibole, a sample containing 2% tremolite in talc was mixed with pure talc to make mixtures containing very low percentage values of tremolite. For example, sample A (Fig. 3) consisting of 0.06% tremolite was made by weighing 58.9 mg of pure talc with 1.7 mg of talc containing 2% tremolite (1.7 mg/60.6 mg × 2% = 0.06%). It is not necessary to make a homogeneous mixture since the entire sample was used in the experiment. Also, the talc containing amphibole was put in the tube second in order not to give the amphibole any "head-start" in sinking to the bottom.

The centrifuge method was also tested with a commercial talc. 100 FOV were counted in ten 1-mg samples according to the FDA procedure for amphibole. This was compared with 20 FOV counts on 60-mg centrifuge samples (Fig. 4). The agreement is quite good. The standard deviations were determined in two

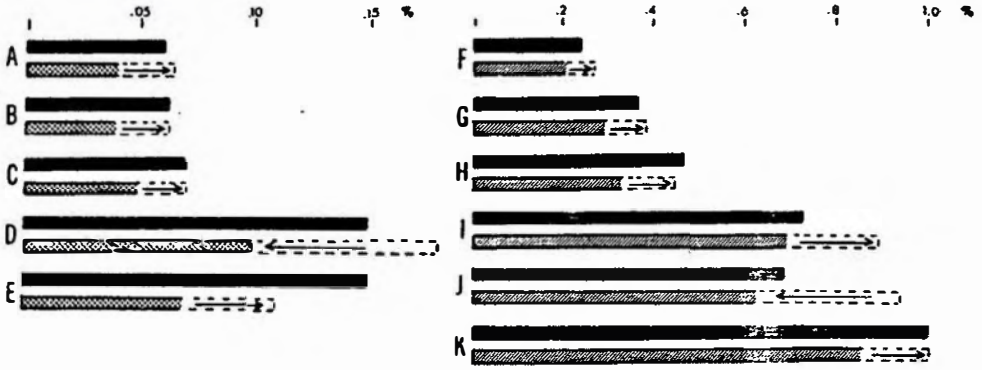

FIGURE 3. Percent tremolite in talc as determined by the centrifuge/optical method (shaded bars) compared with that actually present in experimental mixtures (black bars). The dashed part of the shaded bars indicates +2 SD (right arrow) or −2 SD (left arrow).

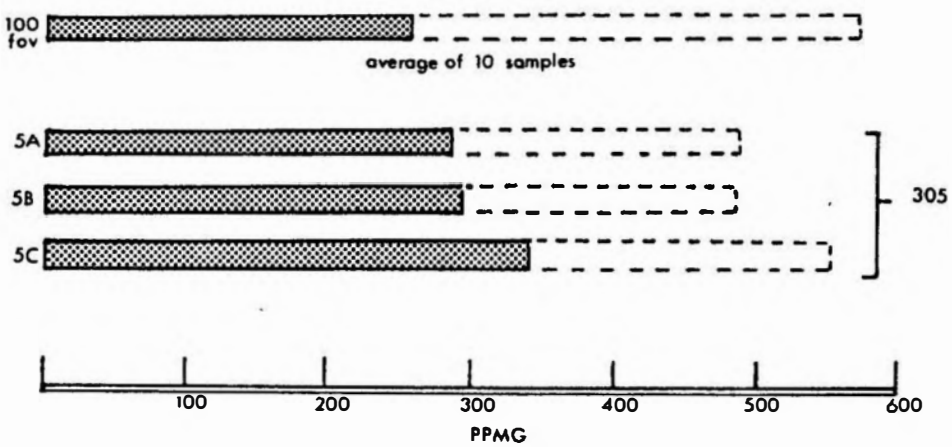

FIGURE 4. Comparison of traditional (100 FOV) count with centrifuge/optical count of same talc. The three lower bars indicate the values in particles/mg obtained by the centrifuge/optical method for three 60-mg samples. The top bar is the average of ten 100 FOV (traditional method). The dashed part of all the bars is +2 SD.

ways: for the traditional method by calculating in the usual way from multiple analyses and for the centrifuge method by means of the Poisson distribution from single counts. Standard deviations are high for the centrifuge method because of the very few particles counted. These could be decreased by making a larger count, but isnce the purpose of the study was to find a reasonably rapid method of monitoring amphibole content of talcs, larger counts were not generally made.

Results

High-grade talc products from five deposits in Montana, three in Vermont, and one each in North Carolina and Alabama were examined using the centrifuge/optical method. In addition, four talcs from outside the U.S. but available in the U.S. market were included in this study. Talcs from other districts in the U.S. were examined, but these talcs had grades with less stringent requirements and are not included in this report.

Results of particle counts are shown in Table 1. The FDA has equated 0.1% with 1000 particles per milligram. In order for amphibole particle content to be less than 0.1%, 20 or less particles must be observed in 20 FOV (5). Since all were well below this value, more extensive counts were not generally made.

It should be borne in mind that the 0.1% indicated is percent by count and not percent by weight or volume. The question of the validity of this relation has been considered (5). Briefly, the relation implies (1000 amphibole particles)/(1,000,000 total particles). Counts of total particles per milligram of talc have shown that 1 million particles per milligram of talc is a low value. Most show at least 2 to 3 times this number. The only exception was a baby powder with very large flakes which showed 0.4 to 0.8 million particles per milligram. It was not clear, however, whether this was a true value or due to the problem of counting where large, flakey particles could potentially hide other particles even in the most carefully prepared samples. Using 1000 particles/mg = 0.1% would, in most samples, give a percentage value on the high side and in this sense be a conservative answer.

The counts shown in Table 1 were made of regulatory fibers i.e., aspect ratio > 3:1. In some samples there were as many or more nonregulatory particles of amphibole as regulatory fibers. The shape of the amphibole varies greatly and seems to be highly characteristic of each deposit. In Table 1, the particles having aspect ratios less than 6:1 are designated cleavages and prismatic pieces. Those greater than 6:1 and less than 15:1 are labeled

Table 1. Counts of regulatory fibers in processed talcs.

Sample	Counts, particles/mg	SD	Particle shapes	Particles/FOV[a]
A	38	25	Cleavages	3/100
B	ND[b]			0/20
C	ND			0/20
D	< 25[c]		Cleavages	0/20
E	ND			0/20
F	ND			0/20
G	ND			0/20
H	17	17	Cleavages and needles	2/20
I	226	59	Needles and fibers	17/20[d]
	283	100	Needles and fibers	8/20
	291	98	Needles and fibers	9/20
	341	108	Needles and fibers	10/20
	102	51	Needles and fibers	3/20
J	25	14	Cleavages	1/20
	27	27	Cleavages	3/20
K	25	25	Cleavages	1/20
L	< 10[c]		Needles	0/20
M	39	21	Cleavages and fibers	4/20
N	25	17	Prismatic pieces	3/20
O	ND			0/20

[a]FOV, fields of view.
[b]ND, none detected.
[c]No particles seen during a 20 FOV count, but some particles could be seen during a random scan of the filter. Value shown is the lower limit of detection.
[d]Large sample used for this analysis (305 mg).

"needles." The remainder, which are greater than 15:1, are labeled "fibers." Whereas in many samples only a few particles were counted as shown in the right-hand column of Table 1, it should be remembered that even if only one particle was present in 20 FOV that about 300 were present on the slide. Because of the low interference by talc particles, these were seen so that it was easy to get a sense of the general particle shape.

The shape distribution of particles for several samples was determined. Figure 5 shows a photograph of a particle of tremolite in sample *I*. The particle is composed of fibrils. The length and width of 100 amphibole particles in this talc were measured. The resulting distribution of aspect ratios is shown in Figure 6. The results when compared with the aspect ratios determinded for tremolite asbestos with SEM by Campbell et al. (*7*) show sample *I* has a distribution similar to asbestos. Sample *M* was analyzed in the same way (Figs. 6 and 7). The graph of aspect ratio verses percent is compared with Campbell's results for nonfibrous tremolite. The similarity of the curves indicates that the tremolite in this talc is of the nonfibrous type.

Because the fractions produced by centrifuge are not generally pure after a single spin-down, a sample containing a variety of particle shapes was tested to see if the aspect ratio distribution results become biased in favor of larger, chunky grains (low aspect ratio) over small, long grains (high aspect ratio). The sample used contained 6.5% tremolite, a sufficient quantity that the traditional optical method could be used to compare with the centifuged sample. The resulting aspect ratio distribution curves (Fig. 8) do not show significant differences. With the traditional method, 69% of the amphibole particles have an aspect ratio of 3:1 or greater, whereas for the centrifuged samples the value is 64%, a variation which is not significant. The differences shown for 5:1 and 10:1 are probably due to the limited number of particles measured, in this test 100 particles in each sample.

Despite the similarity of the curves, the mean length and mean width of the amphibole particles measured using the centrifuge method are greater than those obtained using the traditional method (Table 2). Analysis of size distribution indicates that the proportion larger than 15 μm is greater in the centrifuged sample. This difference in dimension distribution does not appear, however, to affect the aspect ratio distribution. Other investigators have found that as particles increase in length, the aspect ratio shifts to higher values (*8,9*). This applies to both asbestos and nonasbestiform amphiboles, so presumably the effect of centrifuging down longer particles would be to force the aspect ratio distribution peak to higher values.

Discussion

The high-grade talc powders are uniformly low in amphibole content. Counts obtained were 0 to 341 particles/mg. Indeed, talc from some districts appears to be completely free of such minerals. In those containing amphibole minerals there are two distinct types: cleavage type and asbestos type. These two types show distinctly different aspect ratio distributions as demonstrated in Figure 6 (samples *I* and *M*). The aspect ratio difference probably accounts in a large part for the higher particle count per milligram of sample *I* compared with the others which show cleavage fragments. It is easy to see that the number of particles showing greater than 3:1 aspect ratio would be greater in the former case even if the total number of particles of amphibole were equal. This observation reinforces the original decision to count particles visually rather than attempting to use X-ray diffraction. It is not wise to try to convert information on dimensions to percent by weight or volume because a few very large particles can drastically affect the resulting value. Campbell et al. (*8*) discuss this in some detail.

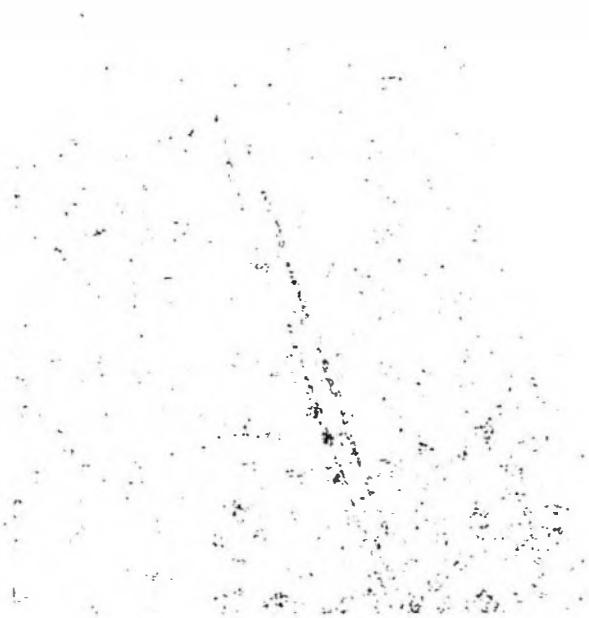

FIGURE 5. Particle of amphibole in centrifuged sample *I*. Width of view 0.07 mm and 1.584 refractive index liquid. Particle is on a membrane filter.

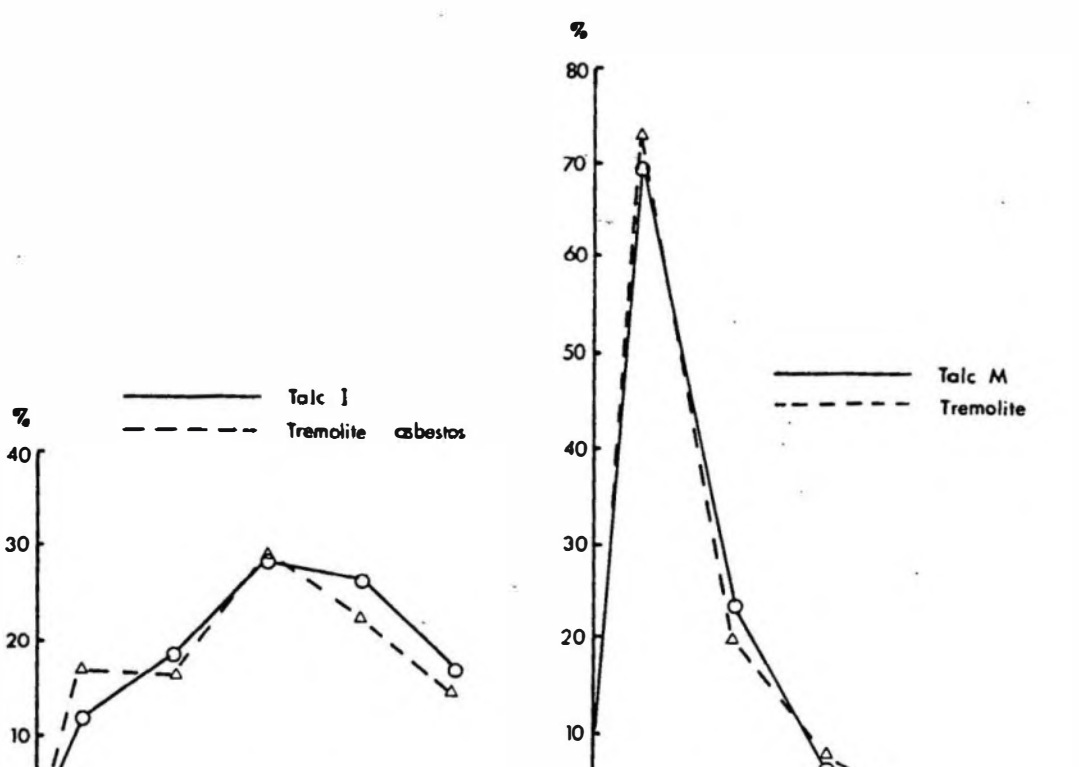

FIGURE 6. Percent amphiboles in each aspect ratio group for talc sample *I* (left) and *M* (right) compared with tremolite asbestos (*7*) and tremolite (nonasbestiform) (*7*).

FIGURE 7. Particle of amphibole in centrifuged sample *M*. Width of view 0.07 mm and 1.584 refractive index liquid. Particle is on a membrane filter.

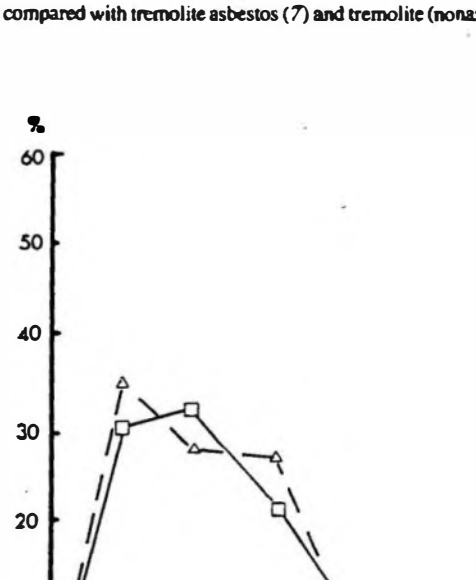

FIGURE 8. Percent amphiboles in each aspect group for a sample handled in two ways: solid line shows results using traditional method and dashed line shows results using centrifuge method. Dimensions of 100 particles measured for each curve.

Table 2. Summary of size and aspect ratio data used to construct Figure 8.

	Size, %		
Method	5–10 μm	10–15 μm	≥ 15 μm
Traditional	57	26	16
Centrifuge	33	27	38
	Mean length, μm	Mean width, μm	Mean aspect ratio, μm
Traditional	12.5	3.0	4.4
Centrifuge	17.5	4.7	4.6

Further, the results from this study demonstrate the utility of the centrifuge method not only for obtaining a count of particles, but also for obtaining information on the shape of particles in a population. It should be emphasized that the aspect ratio curves determined for samples *I* and *M* would have been virtually impossible to obtain using the FDA procedure. The determination would have required examining over 3000 FOV. As indicated previously, many talc flakes on edge appear to be fibers and must be examined during such a scan, making the whole job impossibly tedious.

Finally, even in those cases where one may wish to use the standard 100 FOV count, the centrifuge method offers a way to screen samples between those times when a more lengthy count is made, and it permits a double check of values so determined. In addition, the tendency to bring down a disproportional number of larger particles has the advantage that with true asbestiform amphiboles one generally sees some particles showing bundles of fibrils which removes any doubt about the nature of the amphibole.

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A – Italian

B – Montana – Willow Creek

C – Montana (Beaverhead?) Pfizer

D – North Carolina

E – Alabama

F – Montana – Willow Creek

G – Montana Floated at Alabama (Pfizer) Belmont?

H – Italian

I – Windsor – J&J JBP

J, K, L, M — other VT deposits – M = Troy

N – Timmins Ontario – Steetly

O – Willow Creek – Montana Talc Co.

Exhibit M

11/33.02

CONFIDENTIAL

NEW REAGENT SYSTEMS -

PLANT TRIAL AT

WINDSOR MINERALS INC.

G. LEE

↓

WAA

RSR

EXHIBIT M

INTRODUCTION:

Windsor Minerals has been actively engaged in a froth floatation research program for the past year and a half. This program has developed two floatation reagent systems which offer substantial advantages to the Windsor floatation process from an economic, purity and potential health hazard point of view.

In the absence of known deleterious effects attributable to these reagent systems and in response to the favorable results in testing performed by Baby Products Research in New Brunswick, Windsor Minerals scheduled a plant trial as a guide for establishment of the product and process parameters under actual operating conditions.

The plant trial was conducted on January 29, 1974. During this trial cosmetic grade talcs were produced using both new reagent systems, i.e., n-butyl alcohol and n-bytyl alcohol-citric acid in combination.

The following report will deal with the product and process parameters of the trial as determined by Windsor Minerals and outside consultants.

CONCLUSIONS

1. A reduction in total acid soluble materials was attributable to the new reagent systems. The magnitude of the reduction was 23% for n-butanol and 28% for n-butanol-citric acid.

2. A similar decrease in "magnesite' levels was effected. The decrease was 23% using n-butanol and 30% for the n-butanol-citric acid combination.

3. A color (reflectance) increase of 1.0 units was effected while using n-butanol; an increase of 1.4 units was attributed to the n-butanol-citric acid system.

4. Chrysotile fiber supression was indicated while using the n-butanol-citric acid system.

5. Talc recoveries were higher while using the new reagent systems. The increases were 17% for n-butanol and 19% for n-butanol-citric acid.

6. The chlorite content of the floated product was reduced 14% while using n-butanol and 15% while using n-butanol-citric acid.

7. A substantial decrease in the bulk density of the product was noted while using the new reagent systems. The decreases were 2.06 lb/ft^3 and 2.82 lb/ft^3 respectively for the n-butanol and n-butanol-citric acid systems, corresponding to decreases on a percentage basis of 8.4% and 13.0%.

8. The platy nature of the talc product was found to be unchanged by the use of the new reagent systems.

9. The pH of n-butanol-citric acid floated talc was significantly closer to neutrality than current production. The decrease in alkalinity was measured to be 1.25 pH units for material made during the plant trial.

10. Particle size distribution profiles were similar for materials floated with Ultrawet D.S., n-butanol, and n-butanol-citric acid.

11. Optical microscopy indicates a high degree of similarity with relation to the size and shape factors of materials produced during the plant trial.

Minor differences were noted with respect to talc shards and rolled edges. The product differences, however, correspond quite closely with differences found in the ores from which the products were beneficiated.

OPERATIONAL DESCRIPTION

At 4:30 AM on January 29, 1974, immediately prior to the plant trial using the n-butanol based reagent systems, 1000 pounds of Ultrawet D.S. floated talc was collected and packaged in 4 fiber drums. At the same time a representative ore sample was collected. These materials were used as a reference for the plant trial products.

The floatation circuit was then purged for 3 hours to remove the residual Ultrawet, after which n-butyl alcohol was added at a rate of 1.08 liters per ton of floatation feed.

Sampling was begun after 30 minutes and continued on a 30 minute basis thereafter. The samples were immediately analyzed by the Windsor Minerals Q.A. Laboratory.

After establishing that equilibrium conditions had been reached in the floatation circuitry a 1000 pound sample of finished product was taken and stored in fiber drums for further studies.

Following collection of the n-butanol floated product, citric acid was added to the circuit at a rate of 4 pounds of citric acid per ton of floatation feed, while maintaining the n-butanol additions as before. When the circuit was judged to have reached equilibrium conditions based upon the analytical results, another 1000 pound sample of finished product was taken and stored in fiber drums, also for future studies.

0.2%

SAMPLING

Production sampling for quality assurance purposes was begun at 9:30 AM on 1/29/74 and continued at half hour intervals for the duration of the plant trial. The following table lists the materials sampled and the analyses performed during the trial.

Table 1 Quality Assurance Analytical Schedule

Material Sampled	Sample Qty.	% Acid Insolubles	Color Reflectance	pH	-325 Mesh Screen	Bulk Density	% Magnesite
Ore	500g	X	X		X		
Tailings	250cc	X					
Cleaner Concentrate	500cc	X	X	X			
Product	500g	X	X	X	X	X	X

Composite ore and tailings samples were collected 30 minutes prior to and during each product collection. A product composite for each reagent system used was also obtained from the material packaged in fiber drums.

The composites were then used for the development of analytical data for comparative purposes in assessing the effects of the reagent systems upon the process and resulting products.

EXPERIMENTAL & RESULTS

Table 2 displays a compilation of Quality Assurance data obtained during the test run. The results were used for circuit control, establishment of operational parameters, and talc recovery calculations.

Product composite samples representing materials made with Ultrawet D.S., n-butanol and n-butanol-citric acid were analyzed in accordance with our standard certification procedures. These results, in the form of a standard laboratory report are given in Tables 3, 4 and 5. The data in these tables confirms that the new reagent systems provide substantially improved products in the following categories:

1. total acid solubles
2. magnesite
3. color
4. bulk density
5. pH

There were no specification categories in which a decrease in product qualities were observed.

Table 6 provides the trial results in terms of talc recovery.

Talc recovery was calculated using the relationship:

$$\% \text{ Recovery} = 100 \frac{(H-T)}{(C-T)} \times \frac{C}{H}$$

where:

H = % acid insoluble content of ore

T = % acid insoluble content of tailings

C = % acid insoluble content of cleaner concentrate

Recoveries were derived by obtaining mean acid insoluble values for ore, tailings, and cleaner concentrates from Table 2 for the time period during which the specified reagent was used. These values were compared to the 8 hour production shift immediately preceding the reagent trial during which time Ultrawet D.S. was the floatation reagent. It is apparent from Table 6 that a substantially higher recovery is afforded by the use of n-butanol based floatation systems.

Particle size measurements were performed by two methods; sedimentation velocity using the Andreasen Sedimentation Pipette and by actual optical measurement using the TIMC Image Analyzing system. The results are given in Tables 7-12 and graphically displayed in Figures 1-9.

The particle size distribution profiles indicate the similarity of the products within the framework of the technique used for measurement. However, we have noted and confirmed that differences between the techniques and the values obtained via the techniques do exist. It has been our experience that the direct measurement of particle size and shape which is possible with the Image Analyzing method is a superior determination to the indirect measurements made by the sedimentation method.

On this basis,potential benefit is indicated in that the optical measuring technique has verified a lower fine particle content reporting in the finished product when using the alcohol based systems, particularly the n-butanol-citric acid system. This fact has been confirmed by Walter C. McCrone Associates who have reported the same conclusions based on their optical studies.

Mineralogical examinations for detection of amphiboles were performed by Dr. R. Reynolds at Dartmouth College on the composite ore and product samples. The results for the Ultrawet D.S., n-butanol and n-butanol-citric acid floated products are given in Table 13 and Attachment C, titled "Mineralogy of Ores, Product and Mill Tails Re Different Floatation Reagents".

There were no significant differences with respect to the amphibole content in the test products. The detected amphibole minerals did not appear in a fibrous form in any of the product samples.

Mineralogical analyses using X-ray diffraction techniques were performed on the composite ore, tailings and talc products during each segment of the reagent trial periods. This work was performed by Dr. Reynolds; the results are given in Table 14 and Figure 10.

The gross mineralogical content of the three ore samples were essentially the same.

Analysis of the finished products by X-ray techniques indicate a substantial reduction in chlorite content attributable to the alcohol based systems.

Analysis of the tailings resulting from the use of the three different reagent systems by X-ray diffraction identifies a profound difference in the mineralogical composition. As shown in Figure 10, talc peaks in the alcohol based system tails are roughly one tenth the intensity as found in the Ultrawet system tails. Optical microscopic examination of the tailing fractions from the alcohol based systems also indicates that the small quantity of talc present is essentially all a blocky or non-platy variety. These results confirm the substantial talc recovery differences between the Ultrawet and n-butanol based systems which had been independently determined by chemical analyses.

Asbestiform analyses were performed by Walter C. McCrone Associates by means of transmission electron microscopy and electron diffraction techniques. Their report is found in attachment "A". An abstract of their findings is given in Table 15. Quantitative treatment of these results is questionable due to the extremely low chrysotile levels present, however depression of chrysotile through the use of citric acid in combination with n-butanol is indicated. To better quantify the depressive affects of citric acid upon magnesium surfaced asbestiforms, Attachment "B" titled "Asbestiform depression through the use of new floatation reagent systems" is included to provide details of an earlier study in this area.

SUMMARY & REMARKS

The results of the plant trials using n-butanol and n-butanol-citric acid reagent systems as compared to the presently used Ultrawet D.S. system has indicated a marked superiority of these new systems.

The use of citric acid in the depression of chrysotile asbestos and other mineral species has been developed at Windsor Minerals in response to the potential need for a means to exclude extremely low levels of these contaminants from the finished product of the beneficiation process.

The use of these systems is strongly urged by this writer, to provide the protection against what are currently considered to be

materials presenting a severe health hazard and are potentially present in all talc ores in use at this time.

In closing, based on Windsor's knowledge of the physical chemistry of talc, and upon the results of all work performed to date, it is our strong belief that the use of these new reagent systems will not alter the salient consumer properties of the raw material supply or the finished baby powder sold under the Johnson and Johnson name.

Vernon Zeitz

Vernon Zeitz
Manager, Researsh and Development
Windsor Minerals Inc.
5/14/74

Table 2

QUALITY ASSURANCE SAMPLING TEST RESULTS

TIME	SAMPLE DESIGNATION	ACID INSOLUBLES (%)	COLOR REFLECTANCE (%)	pH	-325 m SCREEN (%)	BULK DENSITY ($lb./ft.^3$)	MAGNESIT (%)
9:30	ORE	58.98	73.4		90.93		
	TAILINGS	28.32					
	CLEANER CONCENTRATE	99.05	88.0	7.57			
	PRODUCT	99.08	87.2	8.06	89.07	24.61	.54
10:00	ORE	62.15	73.8		90.40		
	TAILINGS	25.84					
	CLEANER CONCENTRATE	98.94	87.3	7.78			
	PRODUCT	99.18	87.4	7.92	90.47	23.93	.54
10:30	ORE	64.80	74.6		90.05		
	TAILINGS	24.95					
	CLEANER CONCENTRATE	98.94	87.1	7.79			
	PRODUCT	98.83	87.3	7.92	89.70	23.20	.58
11:00	ORE	61.62	74.8		90.09		
	TAILINGS	26.26					
	CLEANER CONCENTRATE	98.80	87.3	7.81			
	PRODUCT	98.85	87.0	7.99	89.93	23.34	.64
11:30	ORE	65.79	74.8		89.86		
	TAILINGS	26.40					
	CLEANER CONCENTRATE	98.76	87.2	8.42			
	PRODUCT	98.86	87.4	8.44	89.45	23.04	.71
12:00	ORE	65.02	74.4		90.38		
	TAILINGS	28.86					
	CLEANER CONCENTRATE	98.97	87.3	8.19			
	PRODUCT	98.75	87.2	8.10	90.47	22.63	.71

Table 2 Continued

TIME	SAMPLE DESIGNATION	ACID INSOLUBLES (%)	COLOR REFLECTANCE (%)	pH	-325 m SCREEN (%)	BULK DENSITY (lb./ft.^{3})	MAGNESITE (%)
12:30	ORE	66.28	74.0		89.15		
	TAILINGS	24.13					
	CLEANER CONCENTRATE	98.54	86.3	8.19			
	PRODUCT	98.60	86.7	8.10	88.92	23.32	.64
13:00	ORE	63.67	75.0		90.10		
	TAILINGS	28.82					
	CLEANER CONCENTRATE	98.49	86.4	7.89			
	PRODUCT	98.40	86.4	7.81	90.84	22.10	.71
13:30	ORE	64.27	75.3		90.01		
	TAILINGS	23.37					
	CLEANER CONCENTRATE	98.56	86.9	6.38			
	PRODUCT	98.47	87.2	7.42	91.22	21.30	.71
14:00	ORE	61.17	75,3		90.66		
	TAILINGS	26.05					
	CLEANER CONCENTRATE	98.78	87.3	5.28			
	PRODUCT	98.67	87.3	7.01	91.29	21.11	.59
14:30	ORE	57.96	75.3		89.92		
	TAILINGS	24.27					
	CLEANER CONCENTRATE	98.67	87.1	4.64			
	PRODUCT	98.52	87.2	7.22	90.01	21.41	.61
15:00	ORE	61.42	75.2		89.62		
	TAILINGS	23.69					
	CLEANER CONCENTRATE	98.69	87.5	4.38			
	PRODUCT	98.54	87.2	7.10	91.59	21.37	.57

Table 2 Continued

TIME	SAMPLE DESIGNATION	ACID INSOLUBLES (%)	COLOR REFLECTANCE (%)	pH	-325 m SCREEN (%)	BULK DENSITY ($lb./ft.^3$)	MAGNESI (%)
15:30	ORE	62.63	75.2		89.75		
	TAILINGS	22.38					
	CLEANER CONCENTRATE	98.86	87.9	4.38			
	PRODUCT	98.66	87.5	6.92	90.37	21.56	.53
	ORE						
	TAILINGS						
	CLEANER CONCENTRATE						
	PRODUCT						
	ORE						
	TAILINGS						
	CLEANER CONCENTRATE						
	PRODUCT						
	ORE						
	TAILINGS						
	CLEANER CONCENTRATE						
	PRODUCT						
	ORE						
	TAILINGS						
	CLEANER CONCENTRATE						
	PRODUCT						
	ORE						
	TAILINGS						
	CLEANER CONCENTRATE						

Table 3

LABORATORY REPORT

Date Produced 1/29/74

Product or Grade "66 U"

Sample No. or Description Ultrawet D.S. floated product

TEST	FINDINGS	SPECIFIED
MOISTURE	.02	N.M.T. 0.15%
TOTAL ACID SOLUBLE	1.72	N.M.T. 2.0%
MAGNESITE ($MgCO_3$)	.94	N.M.T. 1.10%
COLOR	86.0	WHITE (BY STANDARD)
BULK DENSITY	24.46	20.5 to 25.5 $lbs./ft.^3$
COMPACTION		
MAX. VOLUME	146 cc	
MIN. VOLUME	82 cc	
AVERAGE VOLUME	114 cc	
SCREENS - 60	100.00	100%
- 100	100.00	N.L.T. 99.7%
- 200	98.66	N.L.T. 98.5%
- 325	85.55	
TRACE ELEMENTS		
ARSENIC	.13	N.M.T. 2ppm.
HEAVY METALS	less than 10	N.M.T. 10 ppm.
WATER SOLUBLE IRON	pass test	PASS TEST
MICROSCOPIC EXAMINATION	pass test	PASS TEST
pH	8.89	

Table 4

LABORATORY REPORT

Date Produced 1/29/74

Product or Grade "66 A"

Sample No. or Description N-butanol floated product

TEST	FINDINGS	SPECIFIED
MOISTURE	.03	N.M.T. 0.15%
TOTAL ACID SOLUBLE	1.32	N.M.T. 2.0%
MAGNESITE ($MgCO_3$)	.72	N.M.T. 1.10%
COLOR	87.0	WHITE (BY STANDARD)
BULK DENSITY	22.40	20.5 to 25.5 lbs./ft.^{3}
COMPACTION		
MAX. VOLUME	130 cc	
MIN. VOLUME	94 cc	
AVERAGE VOLUME	112 cc	
SCREENS - 60	100.00	100%
- 100	100.00	N.L.T. 99.7%
- 200	98.99	N.L.T. 98.5%
- 325	91.27	
TRACE ELEMENTS		
ARSENIC	.07	N.M.T. 2ppm.
HEAVY METALS	less than 10	N.M.T. 10 ppm.
WATER SOLUBLE IRON	pass test	PASS TEST
MICROSCOPIC EXAMINATION	pass test	PASS TEST
pH	8.47	

Table 5

LABORATORY REPORT

Date Produced 1/29/74

Product or Grade "66 AC"

Sample No. or Description N-butanol, citric acid floated product

TEST	FINDINGS	SPECIFIED
MOISTURE	.02	N.M.T. 0.15%
TOTAL ACID SOLUBLE	1.23	N.M.T. 2.0%
MAGNESITE ($MgCO_3$)	.66	N.M.T. 1.10%
COLOR	87.4	WHITE (BY STANDARD)
BULK DENSITY	21.64	20.5 to 25.5 lbs./ft.^{3}
COMPACTION		
MAX. VOLUME	140 cc	
MIN. VOLUME	100 cc	
AVERAGE VOLUME	120 cc	
SCREENS - 60	100.00	100%
- 100	100.00	N.L.T. 99.7%
- 200	99.15	N.L.T. 98.5%
- 325	91.97	
TRACE ELEMENTS		
ARSENIC	.17	N.M.T. 2ppm.
HEAVY METALS	less than 10	N.M.T. 10 ppm.
WATER SOLUBLE IRON	pass test	PASS TEST
MICROSCOPIC EXAMINATION	pass test	PASS TEST
pH	7.64	